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(12) Patent:

(11) CA 878423

(54) PARTICULATE COMPOSITIONS SUITABLE FOR USE IN MEAT CURING
AND PROCESS FOR PREPARING THE SAME

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ABSTRACT:

CLAIMS: [Show all claims](#)

*** Note: Data on abstracts and claims is shown in the official language in which it was submitted.

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The present invention relates to new and unobvious compositions of matter suitable for use, for example, in meat curing media, and methods for preparing such compositions.

Further, this invention relates to an improved,
5 granular, compacted, quick-dissolving, free-flowing non-caking, substantially chemically uniform, alkali metal tripolyphosphate-alkali metal hydroxide composition. The alkali metal tripolyphosphate constituent is present in a substantially non-reverted form.

10 Such compositions are suitable for use, for example, in a pickle solution or liquor with other materials such as sodium nitrite and/or sodium nitrate, sodium chloride, sugar, sodium erythorbate, and other edible materials.

Still further, this invention relates to methods of
15 making such compositions using controlled compaction, temperature and water (e.g., moisture or humidity) conditions.

The present invention provides, among other things, compositions capable, when used in a pickle liquor, of fixing the red color of meat products, as well as adjusting the pH of
20 meat products upward to improve the water-binding capacity of meat, and the emulsifying properties of comminuted meat,

Fresh meat normally has a pH in the range between 5.5 and 6.2, largely because of its content of lactic acid formed at slaughter by the break-down of blood and muscle sugar, namely, glycogen. The arterial and/or muscular pumping and injection of curing salt solution or pickle liquor into meat and increasing the pH of the meat toward a pH of 7 are well known. The meat may also be later immersed in a pickle liquor. It is also known that when certain salts of an acid of phosphorus, *

such as alkali metal phosphates, are included in such treatment, the water-binding capacity of meat, particularly whole meat or canned meat products, is increased and in cooking such treated meat there is less shrinkage of the meat with attendant
5 purging of juices. It is also known that the phosphate salts function in this manner in the presence or in the absence of other edible materials such as exemplified by sodium chloride, sugar, sodium nitrite, sodium nitrate, and sodium erythorbate.

Numerous curing compositions have been prepared for
10 use in improving the color, taste, and shrinkage (e.g., during curing, storage, or cooking) of meat products such as beef, pork, mutton, hams (both smoked and canned), picnics, loins, and various sausages such as frankfurters, bologna, and the like. In practice, the alkali metal phosphates which have been
15 available for use as components of meat-curing media are sodium phosphates having an Na_2O to P_2O_5 molar ratio of about 1.7:1. Such alkali metal phosphates have a pH in a 1 percent aqueous solution range, on the order of about 9.8. The increase in the pH of meat inhibits shrinkage of meat products when such phos-
20 phates are injected in the arteries and/or muscles of the meat, or otherwise contacted with the meat. When the pH of the meat is increased above the isoelectric point of the meat proteins, the water-binding capacity of the meat is increased.

In actual practice, the selection of the alkali metal
25 phosphate salt is limited by the solubility characteristics of the phosphate in the pickle liquor, with the sodium chloride content limiting the solubility of certain effective phosphate salts. The effectiveness of the phosphate salts involves the alkali reserve which elevates the pH of the meat toward 7. For

instance, tetrasodium pyrophosphate is more effective weight for weight than sodium tripolyphosphate with respect to water-binding power, but its limited solubility does not permit the dissolving of a desired effective quantity of it in sodium
5 chloride brine to be introduced into the meat.

The use of high concentrations of tetrasodium pyrophosphate, aside from its poor solubility, is objectionable to packers who use pickle liquor made of certain commercial grades of sodium chloride which contain mineral impurities. Calcium
10 ions, for example, react with the tetrasodium pyrophosphate and form precipitates which settle as sludge in the bottom of storage tanks. The poor solubility of tetrasodium pyrophosphate is exemplary of one of the problems in compounding pickle liquors.

Alkali metal tripolyphosphate is commonly used in
15 pickle liquors used for treating meat products because of its good solubility characteristics, its ability to increase the pH of the meat toward a pH of 7, and its ability to improve the water-binding capacity of the meat and thereby induce less shrinkage of the meat with attendant purging of juices during
20 cooking. The ability of alkali metal tripolyphosphate to reduce shrinkage of meat is related in some measure upon an increase in pH of the meat.

Although alkali metal tripolyphosphate, such as sodium tripolyphosphate, is generally regarded as being highly
25 soluble in aqueous pickle liquor, the pH imparted by it to the pickle liquor is not sufficiently high for all purposes. Thus, when sodium tripolyphosphate is used, it is desirable also to use an edible, strongly alkaline material to raise the pH of the pickle liquor and meat to the desired pH values.

Government regulations permit one to use up to 27 pounds of alkali metal tripolyphosphate in 50 gallons of pickle liquor that is used as a 10% pump (10 pounds of pickle liquor per 100 pounds of meat) for pickling meat such as ham.

5 When meat such as ham is treated with pickle liquor having relatively high, but lawful amounts, of alkali metal tripolyphosphate, residual phosphate tends to impart an undesirable phosphate taste to the meat, and to cause the migration of salt content, including residual phosphate, from the meat to
10 the surface of the meat in the form of white "snow", thereby imparting an undesirable surface taste and appearance. Therefore, much less than the upper lawful limit is generally used in pickle liquors used for pickling meat such as ham.

 The usage of sodium hydroxide, since it is highly al-
15 kaline, has been frowned upon by the government authorities as a neutralizer in food products, especially meat. The Canadian government, however, has recently permitted the usage of a small amount of sodium hydroxide as a replacement for phosphates in pumping pickle. Further, it has been found that the
20 use of alkali metal hydroxide can cause more shrink in meat such as ham than results from the use of alkali metal tripolyphosphate, despite the fact that the alkali metal hydroxide increases the pH of the meat to a higher level than the alkali metal hydroxide (e.g., see Example 1). Still further, too high
25 a level of alkali metal hydroxide, such as sodium hydroxide, tends to produce, for some persons, a slightly bitter taste in meat such as ham (e.g., see Examples 1, 4, and 6).

 It has been discovered that the use of alkali metal tripolyphosphate and alkali metal hydroxide, in controlled pro-

portions, and the use of controlled levels in treating meat products, reduces the shrinkage of meat products and attendant purging of juices, avoids the bleeding or salting out of phosphate on the surface of the meat, and avoids the undesired
5 taste qualities which can be found when excessive amounts of either of these materials are used in treating meat.

More specifically, it has been found through extensive testing that the combination of alkali metal tripolyphosphate, when used, for example, in pickling hams, is a synergistic combination that can be effectively used to cause less
10 shrinkage and impart a higher pH in the hams than result from the use of alkali metal tripolyphosphate without the alkali metal hydroxide (e.g., see Examples 2 through 6).

Also, the combination can provide better results than
15 are obtained with alkali metal hydroxide alone or alkali metal tripolyphosphate alone. The combination can be used, for example, to produce less shrink in hams than results from the use of alkali metal hydroxide alone (e.g., compare Example 1 with Examples 2 through 6).

20 The use of high levels of alkali metal hydroxide, such as sodium hydroxide, in a pickle liquor, for example, tends to impart a bitter taste to meat and causes a poor utilization of nitrite from the pickle liquor, as evidenced by the presence of residual nitrite in the treated meat, which tends
25 to prevent the nitrite from producing the stable pink or red color in meat as intended (e.g., see Examples 1, 4, and 6). Powdered alkali metal hydroxide alone is also difficult to handle.

The level of alkali metal hydroxide can be kept down

to a desired level by also using alkali metal tripolyphosphate, and certain undesirable and difficult to control properties of alkali metal hydroxide can be alleviated and substantially chemically uniform granules can be made by compacting a substantially uniform blend of alkali metal hydroxide with alkali metal tripolyphosphate (e.g., see Examples 11, 12, 14, 16, 18, and 20). Still further, this combination can be used in quantities that do not produce the surface "snow", or impart either a phosphate taste to the meat or the bitter taste which is or can be associated with the use of high levels of alkali metal hydroxide.

Sodium hydroxide is very hygroscopic. It is this property, especially when combined with its tendency toward dusting, which makes it difficult to control. That is, it is difficult to weigh out an accurate amount of sodium hydroxide since it absorbs water from the air. Further, it is likely to dust after weighing and before its solution in the pickle is achieved, hence the pickle liquor will contain less than the desired amount of sodium hydroxide. Also, the dust is highly irritating to the eyes and skin of humans and has a corrosive effect upon equipment.

It was found that the dusting problem can be alleviated by forming by compaction the alkali metal tripolyphosphate with the alkali metal hydroxide to produce compacted granules, which may be in the form of pellets, comprising bonded particles of each material. Surprisingly, compaction tends to reduce the hygroscopicity problem. Further, compaction of a substantially homogeneous or uniform blend of materials enables one to produce granules that have a substantially uni-

form chemical content for different gradations of size, although the residual fines may require removal, reblending, and compaction.

Several reversion problems were encountered in attempting to compact the alkali metal tripolyphosphate and alkali metal hydroxide.

The presence of moisture with the alkali metal tripolyphosphate and alkali metal hydroxide blend before the combination is added to an aqueous pickle liquor, tends to cause the alkali metal tripolyphosphate to react with the alkali metal hydroxide and revert (e.g., see Examples 7 and 8) to a less soluble phosphate form (i.e., sodium pyrophosphate and sometimes also sodium orthophosphate). Further, if the blend is subjected to an atmosphere having too high a humidity, reversion results (e.g., see Example 21).

Further, if the temperature of the combination reaches too high a level before, during, or after compaction, substantial reversion will occur or be triggered, and by avoiding such conditions, substantial reversion does not occur (e.g. see Examples 9 through 16 and 18 through 20). This means that the amount of effective compaction pressure that is applied to the blend and the length of time during which compaction occurs must be controlled so as to avoid raising the temperature of the blend to an excessive level and causing substantial reversion.

The present invention includes means for minimizing the above difficulties by providing, for example, granules of compacted curing salt compositions, including both substantially non-reverted alkali metal tripolyphosphate (e.g., sodium

tripolyphosphate) and alkali metal hydroxide (e.g., sodium hydroxide), which granules are substantially chemically uniform in different gradations of size, substantially non-hygroscopic, free-flowing, non-caking and quick-dissolving. The granules
5 are less dusty, less hygroscopic, less irritating, and more easily weighed than powdered alkali metal hydroxide alone or a powdered, non-compacted blend of alkali metal hydroxide and alkali metal tripolyphosphate.

The compositions encompassed by our invention may include a major amount of sodium tripolyphosphate and a minor amount of sodium hydroxide. The composition may include the presence of other alkali metal phosphates or blends thereof (e.g., see Example 19). Although potassium tripolyphosphate may be used under some circumstances, it is not preferred since
15 it causes a bitter taste in some meat products. Again, potassium hydroxide can be used; however, it is not preferred because of the more pronounced bitter taste associated with it. If desired, other edible components of the pickle liquor, such as exemplified by sodium nitrite, sodium nitrate, sodium chloride, and the like, may be included with and/or compacted with
20 the sodium tripolyphosphate and sodium hydroxide, provided the sodium tripolyphosphate does not undergo substantial reversion.

The preferred granular, compacted compositions comprise about 5.5 to 30 parts by weight of alkali metal hydroxide and about 94.5 to 70 parts by weight of alkali metal tripolyphosphate. More specifically, the preferred compositions have about 10 to 20 parts by weight of sodium hydroxide and about
25 90 to 80 parts by weight of sodium tripolyphosphate. Particu-

larly good results have been obtained by pumping meat products such as ham with a 10% pump (10 pounds of pickle liquor per 100 pounds of meat product) having about 2 to 3 pounds of sodium hydroxide, preferably about 2 to 2.5 pounds of sodium hydroxide, and about 8 to 32 pounds, preferably about 12 to 18 pounds, of sodium tripolyphosphate.

After the meat is injected with the pickling or curing solution or brine containing the curing salt composition of this invention, the meat may be smoked, cooked, boned, cured, canned, or otherwise treated. For example, ham treated with a meat curing solution or brine containing the curing salt composition may be boned and then placed in a can or mold and cooked. After standing for several days, canned hams so treated have excellent color retention, texture, and protein hydration properties.

It will be obvious to those skilled in the art that the compaction necessary to accomplish the granulation referred to above may be achieved in many ways. It is essential to form granules using compaction, although the granules may be in the form, for example, of pellets, or even a cake which is subsequently crushed, but which results in a free-flowing or non-caking composition. The exact size and shape of the granules is not deemed to be important as long as the resulting product is free-flowing or non-caking under normal storage conditions.

The examples set forth below are presented to illustrate the invention and advantages thereof more fully without any intention of being limited thereby.

Examples 1 through 6, inclusive, show the results obtained by pumping hams with a 10% pump (10 pounds of pickle

liquor per 100 pounds of ham) of aqueous pickle liquors with varying levels of sodium tripolyphosphate and/or sodium hydroxide. They show that pickle liquors having, among other things, the combination of sodium tripolyphosphate and sodium hydroxide
5 produced better results than pickle liquors having either one of these ingredients alone, although, as one increases the level of sodium tripolyphosphate or sodium hydroxide to certain levels, one tends to impart to the hams a detectable phosphate taste, or somewhat of a bitter taste which results from the so-
10 dium hydroxide.

Examples 7 through 16, and 18 through 21, inclusive, show that the presence of excessive moisture or humidity, or the development of too high a temperature in or with the combination before, during, or after compaction, or the use of too
15 much compaction pressure, tends to cause the sodium tripolyphosphate to undergo substantial reversion, and by avoiding such conditions, substantial reversion is obviated.

Example 17 shows the good solubility characteristics of a compacted sodium tripolyphosphate-sodium hydroxide product
20 in a pickle liquor.

Example 19 also shows that sodium tripolyphosphate is more stable in the presence of sodium hydroxide than certain more highly dehydrated phosphates.

These examples are more fully discussed below.

25 Example 1 shows that the pickle liquor having 16 pounds of sodium tripolyphosphate caused substantially less shrink although a lower pH in ham than the pickle liquor having 3.5 pounds of sodium hydroxide, and the latter pickle liquor caused a poorer utilization of nitrite, as evidenced by more

residual nitrite in the ham, and appeared to produce a slight bitterness in ham.

Example 2 shows that the pickle liquor having 8 pounds of sodium tripolyphosphate and 2 pounds of sodium hydroxide caused less shrink in ham than resulted from the utilization of the pickle liquor having 16 pounds of sodium tripolyphosphate. Example 3 also shows that the pickle liquor with 8 pounds of sodium tripolyphosphate and 2 pounds of sodium hydroxide caused less shrink in ham than resulted from the use of the pickle liquor having 16 pounds of sodium tripolyphosphate.

Example 4 shows that the pickle liquor with 8 pounds of sodium tripolyphosphate and 3 pounds of sodium hydroxide produced in ham slight bitterness according to some tasters and caused less shrink than the pickle liquor with 16 pounds of sodium tripolyphosphate.

Example 5 shows that the pickle liquors having (a) 16 pounds of sodium tripolyphosphate and 2 pounds of sodium hydroxide, (b) 8 pounds of sodium tripolyphosphate and 2 pounds of sodium hydroxide, and (c) 16 pounds of sodium tripolyphosphate, in the order listed, were progressively less effective in reducing shrink of ham.

Example 6 shows that the pickle liquor having 16 pounds of sodium tripolyphosphate and 2.5 pounds of sodium hydroxide, and, to a lesser degree, the pickle liquor with 32 pounds of sodium tripolyphosphate and 2 pounds of sodium hydroxide, and, to a still lesser degree, the pickle liquor with 16 pounds of sodium tripolyphosphate and 2 pounds of sodium hydroxide, caused less shrink of ham than the pickle liquor

with 32 pounds of sodium tripolyphosphate; however, the pickle liquor having 16 pounds of sodium tripolyphosphate produced more shrink in ham than was obtained with 32 pounds of sodium tripolyphosphate; some tasters thought that the pickle liquors
5 with the relatively high levels of phosphate, namely, 32 pounds of sodium tripolyphosphate and 2 pounds of sodium hydroxide, and 32 pounds of sodium tripolyphosphate, respectively, produced in ham a phosphate taste; and some tasters thought that the pickle liquor with 16 pounds of sodium tripolyphos-
10 phate and 2.5 pounds of sodium hydroxide appeared to produce a slightly bitter taste.

Example 7 shows that when a moist blend having 8 parts by weight of sodium tripolyphosphate and 2 parts by weight of sodium hydroxide was heated, the sodium tripolyphos-
15 phate substantially reverted.

Example 8 shows that the sodium tripolyphosphate in a moist blend having 500 parts by weight of sodium tripolyphosphate, 42 parts by weight of sodium hydroxide, and 78 parts by weight of water, undergoes reversion that increases with the
20 passage of time. This appears to indicate that moisture induces reversion of the phosphate.

Example 9 shows that sodium tripolyphosphate in a blend of 80% of sodium tripolyphosphate and 20% of sodium hydroxide tends to undergo reversion when it is heated sufficiently to progressively higher temperatures.
25

Example 10 shows that sodium tripolyphosphate in blends having 80% of sodium tripolyphosphate and 20% of sodium hydroxide (no water was added as such, although too high a humidity can cause moisture to be absorbed) tends to undergo

increasing degrees of reversion as the blends were heated sufficiently to progressively higher temperatures.

Example 11 shows that when a blend having 80% of sodium tripolyphosphate and 20% of sodium hydroxide (no water was added as such) was subjected at room temperature to increasing levels of compaction pressure in a Carver press, the level of reversion of the sodium tripolyphosphate tends to increase. Good compacted granules or pellets were obtained and no or little reversion occurred when the blend was compacted with sufficiently low pressures.

Example 12, which is similar to Example 11, also shows that as one increases the amount of compaction pressure above certain levels, particularly if the blend is heated to higher temperatures prior to compaction, the degree of reversion of the sodium tripolyphosphate tends to increase.

Example 13 shows that the development of too much heat prior to compaction and/or excessive compaction pressure tend to cause the generation of heat and substantial reversion of the sodium tripolyphosphate with blends (to which no water was added as such) having either 80% of sodium tripolyphosphate and 20% of sodium hydroxide, or 702 pounds of sodium tripolyphosphate and 167 pounds of sodium hydroxide (see Example 9).

Example 14 shows that by cooling the blend before compaction and by cooling the compaction rolls, a blend of 80% of sodium tripolyphosphate and 20% of sodium hydroxide can be effectively compacted into granules without causing any visible substantial reversion of the sodium tripolyphosphate.

Example 15 shows that if a blend of 80% of sodium tripolyphosphate and 20% of sodium hydroxide is at too high a

temperature prior and/or during compaction, reversion of the sodium tripolyphosphate can be triggered and substantial reversion can occur with the passage of time.

Example 16 shows that a blend (to which no water was added as such) having 8 parts of sodium tripolyphosphate and 2 pounds of sodium hydroxide could be effectively compacted with the compaction rolls of Example 14 without having the phosphate undergo substantial reversion.

Example 17 shows that compacted Sample 3B of Example 14 (80% of sodium tripolyphosphate and 20% of sodium hydroxide) had good solubility characteristics when it was added to a pickle liquor.

Example 18 shows further the need for not subjecting a blend of sodium tripolyphosphate-sodium hydroxide to excessively high temperatures prior to compaction, subjecting the blend to effective compaction pressure without causing an excessively high temperature rise, and maintaining a controlled, not excessively high temperature during storage or packaging of the compacted granules.

Example 19 shows that: blends having non-compacted sodium tripolyphosphate and sodium hydroxide tend to revert as the temperature increases; within the limits tested, the initiation of reversion does not appear to be dependent upon the proportion of sodium hydroxide present, however, if reversion does occur, the extent of the reversion tends to increase with an increase in the proportion of sodium hydroxide; and blends with sodium tripolyphosphate appear to be more heat stable with sodium hydroxide than certain more highly dehydrated phosphates.

Example 20 shows that the compacted blends of Examples 14 (Sample 3B), 15, and 18 exhibited no substantial reversion after being stored for selected periods of time.

Example 21 shows that the sodium tripolyphosphate present in a sodium tripolyphosphate-sodium hydroxide blend undergoes reversion in an atmosphere having too high a humidity.

All parts and percentages expressed in the following examples are on a weight basis unless otherwise specified.

Further, the sodium tripolyphosphate used in the examples was in a powdered, anhydrous form. The sodium hydroxide used was also initially in a powdered form. The blends that were compacted were substantially homogeneous or substantially uniformly blended before compaction.

Example 1

The following pickle liquors were prepared:

Pickle Liquor "A"	
Water	50 gallons (416.5 pounds)
Sodium Chloride	71.25 pounds
20 Cane Sugar	25.00 pounds
Prague Powder	13.75 pounds
90% Sodium Chloride	
4% Sodium Nitrate	
6% Sodium Nitrite	
Sodium Tripolyphosphate	16.00 pounds

 Pickle Liquor "B"

	Water	50 gallons (416.5 pounds)
	Sodium Chloride	71.25 pounds
	Cane Sugar	25.00 pounds
5	Prague Powder	13.75 pounds
	90% Sodium Chloride	
	4% Sodium Nitrate	
	6% Sodium Nitrite	
	Sodium Hydroxide	3.50 pounds

10 It should be noted that Pickle Liquor "A" differs from Pickle Liquor "B" in that the former has 16 pounds of sodium tripolyphosphate, whereas the latter has 3.50 pounds of sodium hydroxide.

15 Three sets of paired hams were pumped with 10% pump (10 pounds of pickle liquor per 100 pounds of ham) so that the left ham from each of the 3 sets was pumped with Pickle Liquor "A" and the right ham of each set was pumped with Pickle Liquor "B".

20 The hams were then rubbed lightly with Superior Bacon Mix (i.e., 70.13% sodium chloride, 28.25% sugar, 1.02% sodium nitrite, and 0.67% sodium nitrate) and held in the cure for 5 days at 45°F., followed by boning, canning, and cooking. The hams were held in the storage cooler at 45°F. for 6 days, after which the cans were opened and the hams were evaluated.

25 The data shown in Table 1 were obtained from the analysis of the pickle liquors and hams.

Table 1

	<u>Pickle Liquor "A"</u>	<u>Pickle Liquor "B"</u>
pH of Pickle Liquor	7.65	11.45
Salometer	85.00°	76.00°
5 Average pH of Finished Ham	6.17	6.45
Average Residual Nitrite in Finished Ham	68.00 ppm.	114.50 ppm.
10 Average Shrink of Three Sets of Paired Hams	10.70%	17.70%

The above data of Table 1 show that Pickle Liquor "A", with 16 pounds of sodium tripolyphosphate, caused substantially less shrink and lower pH than Pickle Liquor "B", with 3.5 pounds of sodium hydroxide, and Pickle Liquor "B" caused a poorer utilization of nitrite as evidenced by the presence of more residual nitrite.

The tests were repeated with 2 different sets of hams, and the data shown in Table 2 were obtained from the analysis of these sets of hams.

20

Table 2

	<u>Pickle Liquor "A"</u>	<u>Pickle Liquor "B"</u>
Average Shrink of Hams	11.2%	16.3%

The data shown in Table 2 corresponds with the data in Table 1.

The hams were sliced and it was noted that the inside color of the hams treated with Pickle Liquor "A" was slightly better than the color of the hams treated with Pickle Liquor "B". After tasting the hams, one of the five tasters thought there was a slight bitterness in the hams treated with Pickle Liquor "B".

Portions of the ham were sliced and packaged in vacuum and the packages were exposed to fluorescent light at 75 to 90 foot candles. After the first day of exposure, the slices showed considerable graying, regardless of whether the hams were treated with Pickle Liquor "A" or Pickle Liquor "B"; however, a slightly grayer color was noticed with regard to the hams treated with Pickle Liquor "B". After one or two days' exposure to the fluorescent light, the original color returned. The color remained after ten days of exposure.

10

Example 2Pickle Liquor "A"

Water	50 gallons (416.5 pounds)
Sodium Chloride	71 pounds, 4 ounces
Cane Sugar	25 pounds
15 Prague Powder	13 pounds, 12 ounces
Sodium Tripolyphosphate	8 pounds
Sodium Hydroxide	2 pounds

Pickle Liquor "B"

Water	50 gallons (416.5 pounds)
20 Sodium Chloride	71 pounds, 4 ounces
Cane Sugar	25 pounds
Prague Powder	13 pounds, 12 ounces
Sodium Tripolyphosphate	16 pounds

It should be noted that Pickle Liquor "A" differs from Pickle Liquor "B" in that the former has 8 pounds of sodium tripolyphosphate and 2 pounds of sodium hydroxide, whereas the latter has 16 pounds of sodium tripolyphosphate.

Three sets of paired hams were pumped with 10% pump so that the right ham from each of the 3 sets was pumped with

Pickle Liquor "A" and the left ham of each set was pumped with Pickle Liquor "B".

The hams were then rubbed lightly with Superior Bacon Mix and held in the cure for 5 days, followed by boning, can-
 5 ning and cooking. The hams were held in the storage cooler for 7 days, after which the cans were opened and the hams were evaluated.

The data shown in Table 3 were obtained.

Table 3

	<u>Pickle Liquor "A"</u>	<u>Pickle Liquor "B"</u>
10 pH of Pickle Liquor	11.20	7.32
Salometer	76.00°	82.00°
pH of Ground Ham	6.35	6.20
Residual Nitrite in		
15 the Ground Ham	91.00 ppm.	74.00 ppm.
Average Shrink	7.73%	8.17%

The hams pumped with Pickle Liquor "A", with 8 pounds of sodium tripolyphosphate and 2 pounds of sodium hydroxide, caused a slightly higher pH, but somewhat lower shrink
 20 than hams pumped with Pickle Liquor "B", with 16 pounds of sodium tripolyphosphate. Pickle Liquor "A" did not produce a bitter taste in the finished ham, which is sometimes associated with sodium hydroxide, and it appeared to produce a somewhat tastier flavor than Pickle Liquor "B".

Example 3

This example is similar to Example 2.

The following pickle liquors were prepared:

Pickle Liquor "A"

	Water	50 gallons (416.5 pounds)
	Sodium Chloride	71.25 pounds
	Cane Sugar	25.00 pounds
5	Prague Powder	13.00 pounds, 12 ounces
	Sodium Tripolyphosphate	8.00 pounds
	Sodium Hydroxide	2.00 pounds

Pickle Liquor "B"

	Water	50 gallons (416.5 pounds)
10	Sodium Chloride	71.25 pounds
	Cane Sugar	25.00 pounds
	Prague Powder	13.00 pounds, 12 ounces
	Sodium Tripolyphosphate	16.00 pounds

It should be noted that Pickle Liquor "A" differs
 15 from Pickle Liquor "B" in that the former has 8 pounds of so-
 dium tripolyphosphate and 2 pounds of sodium hydroxide, whereas
 the latter has 16 pounds of sodium tripolyphosphate.

Two sets of paired hams were pumped with 10% pump so
 that the right ham from each of the 2 sets was pumped with
 20 Pickle Liquor "A" and the left ham of each set was pumped with
 Pickle Liquor "B".

The hams were then rubbed lightly with Superior Bacon
 Mix and held in the cure for 5 days, followed by boning, can-
 ning, and cooking. The hams were held in the storage cooler
 25 for 7 days, after which the cans were opened and the hams were
 evaluated.

The data shown in Table 4 were obtained.

Table 4

	<u>Pickle Liquor "A"</u>	<u>Pickle Liquor "B"</u>
pH of Pickle Liquor	11.80	7.32
Salometer	77.00°	82.00°
5 pH of Ground Ham	6.50	6.20
Residual Nitrite	146.00 ppm.	98.00 ppm.
Average Shrink	7.67%	9.77%

The hams pumped with Pickle Liquor "A", with 8 pounds of sodium tripolyphosphate and 2 pounds of sodium hy-
 10 droxide, did not have a bitter taste, and had less shrink and a somewhat higher pH than the hams treated with Pickle Liquor "B", with 16 pounds of sodium tripolyphosphate.

Example 4

The following pickle liquors were prepared:

15	<u>Pickle Liquor "A"</u>	
	Water	50 gallons (416.5 pounds)
	Sodium Chloride	71 pounds, 4 ounces
	Cane Sugar	25 pounds
	Prague Powder	13 pounds, 12 ounces
20	Sodium Tripolyphosphate	8 pounds
	Sodium Hydroxide	3 pounds
	<u>Pickle Liquor "B"</u>	
	Water	50 gallons (416.5 pounds)
	Sodium Chloride	71 pounds, 4 ounces
25	Cane Sugar	25 pounds
	Prague Powder	13 pounds, 12 ounces
	Sodium Tripolyphosphate	16 pounds

It should be noted that Pickle Liquor "A" differs from Pickle Liquor "B" in that the former has 8 pounds of so-

dium tripolyphosphate and 3 pounds of sodium hydroxide, whereas the latter has 16 pounds of sodium tripolyphosphate.

Three sets of paired hams were pumped with 10% pump so that the right ham from each of the 3 sets was pumped with
5 Pickle Liquor "A" and the left ham of each set was pumped with Pickle Liquor "B".

The hams were then rubbed lightly with Superior Bacon Mix and held in the cure for 5 days, followed by boning, canning, and cooking. The hams were held in the storage cooler
10 for 7 days, after which the cans were opened and the hams were evaluated.

The data shown in Table 5 were obtained.

Table 5

	<u>Pickle Liquor "A"</u>	<u>Pickle Liquor "B"</u>
15 pH of Pickle Liquor	11.80	7.32
Salometer	77.00°	82.00°
pH of Ground Ham	6.35	6.20
Residual Nitrite in the Ground Ham	109.00 ppm.	79.00 ppm.
20 Average Shrink	8.28%	8.70%

In conclusion, Pickle Liquor "A", with 8 pounds of sodium tripolyphosphate and 3 pounds of sodium hydroxide, produced in ham less shrink than Pickle Liquor "B", with 16 pounds of sodium tripolyphosphate. Further, Pickle Liquor "A"
25 produced a slight bitterness according to some of the tasters.

Example 5

The following pickle liquors were prepared:

Pickle Liquor "A"

	Water	50 gallons (416.5 pounds)
	Sodium Chloride	71 pounds, 4 ounces
	Cane Sugar	25 pounds
5	Prague Powder	13 pounds, 12 ounces
	Sodium Tripolyphosphate	16 pounds
	Sodium Hydroxide	2 pounds

Pickle Liquor "B"

	Water	50 gallons (416.5 pounds)
10	Sodium Chloride	71 pounds, 4 ounces
	Cane Sugar	25 pounds
	Prague Powder	13 pounds, 12 ounces
	Sodium Tripolyphosphate	8 pounds
	Sodium Hydroxide	2 pounds

Pickle Liquor "C"

	Water	50 gallons (416.5 pounds)
	Sodium Chloride	71 pounds, 4 ounces
	Cane Sugar	25 pounds
	Prague Powder	13 pounds, 12 ounces
20	Sodium Tripolyphosphate	16 pounds

It should be noted that Pickle Liquors "A" through "C", as shown in Table 6, differ in composition as follows:

Table 6

	<u>Pickle Liquors</u>		
	<u>"A"</u>	<u>"B"</u>	<u>"C"</u>
25			
	Sodium Tripolyphosphate	16 pounds	8 pounds
	Sodium Hydroxide	2 pounds	2 pounds

Three sets of paired hams were pumped with 10% pump so that the left ham from each of the 3 sets was pumped with

Pickle Liquor "C" and the right ham of each set was pumped with Pickle Liquor "A".

Two additional sets of paired hams were pumped with 10% pump so that the left ham from each of the 2 sets was pumped with Pickle Liquor "C" and the right ham of each set of hams was pumped with Pickle Liquor "B"

Thus, the pumping procedure shown in Table 7 was followed.

<u>Table 7</u>			
	<u>Ham Number</u>	<u>Left Ham</u>	<u>Right Ham</u>
10	1	"C"	"A"
	2	"C"	"A"
	3	"C"	"A"
	4	"C"	"B"
15	5	"C"	"B"

The hams were then rubbed lightly with Superior Bacon Mix and held in the cure for 5 days, followed by boning, canning, and cooking. The hams were held in the storage cooler for 7 days, after which the cans were opened and the hams were evaluated.

The average shrinkage data shown in Table 8 were obtained.

<u>Table 8</u>		
	<u>Pickle Liquor "A"</u>	<u>Pickle Liquor "C"</u>
25 Average Shrink	9.36%	11.50%
	<u>Pickle Liquor "B"</u>	<u>Pickle Liquor "C"</u>
Average Shrink	10.39%	10.52%

The hams treated with each of the pickle liquors had comparable color, although Pickle Liquors "A" and "B" appeared

to produce a slightly better color than Pickle Liquor "C". The hams produced with Pickle Liquors "A" and "B" were considered to have excellent taste and were comparable to the hams produced with Pickle Liquor "C".

5 In conclusion, Pickle Liquor "A", in particular, with 16 pounds of sodium tripolyphosphate and 2 pounds of sodium hydroxide, as well as Pickle Liquor "B", with 8 pounds of sodium tripolyphosphate and 2 pounds of sodium hydroxide, produced less shrink than Pickle Liquor "C" with 16 pounds of sodium tripolyphosphate.

Example 6

The following pickle liquors were prepared:

<u>Pickle Liquor "A"</u>	
Water	50 gallons (416.5 pounds)
15 Fine Flake Salt	71 pounds, 4 ounces
Cane Sugar	25 pounds
Prague Powder	13 pounds, 12 ounces
Sodium Tripolyphosphate	32 pounds
Sodium Hydroxide	2 pounds
<u>Pickle Liquor "B"</u>	
20 Water	50 gallons (416.5 pounds)
Fine Flake Salt	71 pounds, 4 ounces
Cane Sugar	25 pounds
Prague Powder	13 pounds, 12 ounces
25 Sodium Tripolyphosphate	16 pounds
Sodium Hydroxide	2 pounds, 8 ounces

Pickle Liquor "C"

	Water	50 gallons (416.5 pounds)
	Fine Flake Salt	71 pounds, 4 ounces
	Cane Sugar	25 pounds
5	Prague Powder	13 pounds, 12 ounces
	Sodium Tripolyphosphate	16 pounds
	Sodium Hydroxide	2 pounds

Pickle Liquor "D"

	Water	50 gallons (416.5 pounds)
10	Fine Flake Salt	71 pounds, 4 ounces
	Cane Sugar	25 pounds
	Prague Powder	13 pounds, 12 ounces
	Sodium Tripolyphosphate	32 pounds

Pickle Liquor "E"

15	Water	50 gallons (416.5 pounds)
	Fine Flake Salt	71 pounds, 4 ounces
	Cane Sugar	25 pounds
	Prague Powder	13 pounds, 12 ounces
	Sodium Tripolyphosphate	16 pounds

20 It should be noted that Pickle Liquors "A" through "E" differ in composition as shown in Table 9 below.

Table 9

		<u>Pickle Liquors</u>				
		<u>"A"</u>	<u>"B"</u>	<u>"C"</u>	<u>"D"</u>	<u>"E"</u>
25	Sodium Tripolyphosphate	32 pounds	16.0 pounds	16 pounds	32 pounds	16 pounds
	Sodium Hydroxide	2 pounds	2.5 pounds	2 pounds	-----	-----

30 Further, these pickle liquors had the characteristics shown in Table 10 below.

Table 10

		<u>Pickle Liquors</u>				
		<u>"A"</u>	<u>"B"</u>	<u>"C"</u>	<u>"D"</u>	<u>"E"</u>
	pH	10.91	10.98	10.9	7.42	7.5
5	Salometer	88.00°	81.00°	81.0°	88.00°	81.0°

Twelve sets of paired hams were pumped with 10% pump so that the right ham from each of the 12 sets was pumped with the same pickle liquor and the left ham of each of 3 different sets of hams was pumped with another, different pickle liquor.

10 More specifically, Pickle Liquor "D" (the right ham) was compared with Pickle Liquors "A", "B", "C", and "E" (the left ham). The liquors that were compared followed the pumping procedure shown in Table 11 below.

Table 11

	<u>Ham Number</u>	<u>Left Ham</u>	<u>Right Ham</u>
15	1	"E"	"D"
	2	"E"	"D"
	3	"E"	"D"
	4	"C"	"D"
20	5	"C"	"D"
	6	"C"	"D"
	7	"B"	"D"
	8	"B"	"D"
	9	"B"	"D"
25	10	"A"	"D"
	11	"A"	"D"
	12	"A"	"D"

The hams were rubbed lightly with Superior Bacon Mix and held in the cure for 4 days, followed by boning, vacuum

30 canning, cooking, and chilling. The hams were held in cooler storage for 6 days, after which the cans were opened and the hams were evaluated.

The average shrink data shown in Table 11A were ob-

tained.

Table 11A

	<u>Pickle Liquor "A"</u>	<u>Pickle Liquor "D"</u>
Average Shrink	9.60%	10.2%
5	<u>Pickle Liquor "B"</u>	<u>Pickle Liquor "D"</u>
Average Shrink	7.80%	9.6%
	<u>Pickle Liquor "C"</u>	<u>Pickle Liquor "D"</u>
Average Shrink	8.08%	8.6%
	<u>Pickle Liquor "E"</u>	<u>Pickle Liquor "D"</u>
10 Average Shrink	10.20%	8.4%

The hams that were treated with Pickle Liquors "A" and "D" having the 32 pounds of sodium tripolyphosphate provided some indication of a phosphate taste to most of the tasters. Some of the tasters thought that the hams treated with Pickle Liquor "B" (16 pounds of sodium tripolyphosphate and 2.5 pounds of sodium hydroxide) had a slightly bitter taste, whereas the hams treated with Pickle Liquor "C" (16 pounds of sodium tripolyphosphate and 2 pounds of sodium hydroxide) had an excellent flavor.

In conclusion, Pickle Liquor "B", in particular, with 16 pounds of sodium tripolyphosphate and 2.5 pounds of sodium hydroxide, and to a somewhat lesser degree, Pickle Liquor "A", with 32 pounds of sodium tripolyphosphate and 2 pounds of sodium hydroxide, and to a still lesser degree, Pickle Liquor "C", with 16 pounds of sodium tripolyphosphate and 2 pounds of sodium hydroxide, produced in ham less shrink than Pickle Liquor "D" with 32 pounds of sodium tripolyphosphate. Pickle Liquor "E", with 16 pounds of sodium tripolyphosphate, produced more shrink in ham than Pickle Liquor "D", with 32 pounds of

sodium tripolyphosphate.

Example 7

The following blend was prepared:

	Sodium Tripolyphosphate	80 gm.
5	Sodium Hydroxide	20 gm.
	Water	50 cc.

The blend was dried over night in an oven at 175°F.

The resulting dried cake was analyzed and it was found that almost all of the sodium tripolyphosphate had reverted to sodium pyrophosphate as determined by paper chromatography and altered titration curves. (In this and other examples below, a 1-gram sample was dissolved in 100 cc. of water and the solution was titrated with 0.1N sulfuric acid to a pH of 3.8.)

These results indicate that the presence of water (e.g., moisture) tends to make a sodium tripolyphosphate-sodium hydroxide blend unstable.

Example 8

A blend of the following materials was prepared:

	Sodium Tripolyphosphate	500 grams
20	Sodium Hydroxide	42 grams
	Water	78 grams

The sodium hydroxide was added as 84 grams of a 50% aqueous solution and 36 grams of additional water were added.

The blend was divided into 8 equal parts and placed in tightly sealed jars at room temperature. The jars were placed in a closed container and stability studies were made to determine the degree of reversion of the sodium tripolyphosphate to sodium ortho- and pyrophosphates.

The free or unreacted sodium hydroxide was determined

in two ways. In the first method a 1-gram sample was extracted with 100 ml. of ethyl alcohol, and the extract was titrated with 0.1N sulfuric acid to a pH of 7.0. This method determines the sodium hydroxide content, since the phosphates, sodium tripolyphosphate and its reversion products (e.g., sodium ortho- and pyrophosphates), are not soluble in alcohol. In the second method, 100 ml. of a 1% aqueous solution of the sample was titrated with 0.1N sulfuric acid to a pH of 10.5. This method is a specific measurement for sodium hydroxide as long as no sodium orthophosphate is present. When the latter begins to appear in significant amounts (as a result of reversion of sodium tripolyphosphate) this method will yield higher results than the first one. The moisture content of the material was determined by drying a sample for 6 hours in a vacuum oven at 50°C. (122°F.). The phosphate composition was determined by paper chromatography using appropriate standards and titration curves as described in Example 7.

Table 12 below shows the analysis of the blend on the day it was prepared.

		<u>Table 12</u>	
		<u>Determined by Analysis</u>	<u>Calculated from Known Ingredients</u>
20	Alcohol-Soluble Sodium Hydroxide	6.20%	6.71%
25	Titratable Sodium Hydroxide	5.60%	-----
	Moisture	11.90%	12.60%
Phosphate Composition			
	Tripolyphosphate	95.00%	-----
30	Pyrophosphate	5.00%	-----
	Orthophosphate	0.00%	-----
	pH (1% solution)	11.61	-----

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Table 13 below shows the analysis of the blend at periodic intervals. The analysis was made using the above referred to methods. The stability of the prepared composition was determined at 3- to 5-day intervals. Fresh jars were
5 opened each time to eliminate the possibility of breakdown due to frequent handling. The results were as shown in Table 13 below.

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Table 13

Length of Storage	pH (1% Solution)	% NaOH (Alcohol-Soluble)	% of Total NaOH (Still Unreacted)	% Titratable Alkalinity (to pH 10.5)	% Phosphate Composition		
					Sodium Tripolyphosphate	Sodium Pyrophosphate	Sodium Orthophosphate
0 days	11.61	6.20	92	5.60	95	5	0
3 days	-----	-----	--	-----	90	5	5
5 days	11.59	4.95	74	4.80	80	10	10
10 days	11.52	4.28	64	4.40	80	10	10
14 days	11.50	3.41	51	4.04	75	20	5
24 days	11.19	1.68	25	3.01	60	30	10

10

The results in Table 13 show that 92% of the calculated amount of free sodium hydroxide can be extracted from the composition on the day of manufacture, and 24 days later, 25% of the free sodium hydroxide remained unreacted. The remaining
 5 75% of free sodium hydroxide reacted with sodium tripolyphosphate, resulting in reversion of the sodium tripolyphosphate to sodium ortho- and pyrophosphates, as evidenced above.

These results also indicate that the presence of water (e.g., moisture) tends to make a sodium tripolyphosphate-
 10 sodium hydroxide blend unstable, particularly with the passage of time.

Example 9

One hundred gram samples of a blend of 80% sodium tripolyphosphate and 20% of sodium hydroxide were placed in
 15 beakers immersed in a heated oil bath and the blends were agitated with a paddle-type stirrer for 30 minutes at the temperatures shown in Table 14. The results were as shown in Table 14 below.

Table 14

20	<u>Temperature of Heating</u>	<u>Reversion of Sodium Tripolyphosphate</u>
	47°C. (116°F.)	0%
	61°C. (140°F.)	10%
	75°C. (167°F.)	50%

25 These results point out the importance of controlling the temperature, otherwise reversion occurs. At a temperature of 167°F., within 30 minutes up to 50% of the sodium tripolyphosphate reverted to sodium ortho- and pyrophosphates as determined by paper chromatography and titration (see Example 7).

Even at 140°F., some reversion occurred. When this same experiment was conducted at 116°F., no reaction occurred between the sodium hydroxide and the sodium tripolyphosphate.

Again, this shows that too high an increase in the
5 temperature of the blend can cause substantial reversion.

Example 10

A blend was made by mixing 80% powdered, anhydrous sodium tripolyphosphate and 20% powdered sodium hydroxide. A major proportion of both ingredients passed through a 100-mesh screen.
10 The samples were placed in a muffle furnace at the temperatures indicated in Table 15 below for 30 minutes, and the degree of reversion of the sodium tripolyphosphate to sodium ortho- and pyrophosphates was determined by paper chromatography and titration (see Example 7).

15

Table 15

	<u>Muffle Temperature</u>	<u>% Moisture Loss From the Sample</u>	<u>Reversion of Sodium Tripolyphosphate</u>
	600°C.	6.0	60%
	500°C.	5.8	40%
20	400°C.	5.2	25%
	300°C.	5.0	20%
	200°C.	3.7	15%
	110°C.	1.7	10%
	Control (No Heat)	---	---

25

The results given in Table 15 above show that 15% reversion occurred when the blend was heated to a temperature of 200°C. and substantial reversion occurs and increases at higher temperatures. The moisture loss by the sample is a product of the reaction in which the sodium tripolyphosphate

reverts to sodium ortho- and pyrophosphates.

The above results show the need of controlling the temperature of blends of sodium tripolyphosphate and sodium hydroxide.

5

Example 11

A blend similar to that used in Example 10 above of 20% sodium hydroxide and 80% sodium tripolyphosphate was prepared. 3.5 Gram samples of the blend were added to a one-inch die and placed in a Carver press having a tube fitted with a one-inch diameter piston. The pellets formed on compaction had a thickness of approximately 1/8 inch. The compaction was carried out at room temperature in the presence of no added moisture. Several pellets were made under each condition of compaction. The results of the compaction are given in Table 16 below. (A good pellet is one that is not fragile.)

Table 16

	<u>Pressure (Psi.)</u>	<u>Observation of Fragility of Compacted Product</u>	<u>Degree of Reversion of Sodium Tripolyphosphate</u>
20	2,000	Poor Pellets	0%
	3,000	Good Pellets	0%
	6,000	Good Pellets	0%
	9,000	Good Pellets	5%
	12,000	Good Pellets	40%

25

The results shown in Table 16 above show that if excessive pressure is applied, even at room temperature, the sodium hydroxide may react with the sodium tripolyphosphate to revert the sodium tripolyphosphate to sodium ortho- and pyrophosphates, as evidenced by paper chromatography and titration

30 (see Example 7).

The respective samples of pellets were broken down in size, and the resulting products which passed through a 5-mesh screen and remained on a 40-mesh screen were stored for 3 weeks at room temperature. No further sign of reversion occurred with those pellets which exhibited no substantial reversion immediately after compaction.

Example 12

A similar experiment was conducted to that set forth in Example 11 in that a blend of 80% of sodium tripolyphosphate and 20% of sodium hydroxide was made. The blend was placed in an oven to a temperature of 110°C. for 2 minutes, which warmed the sample. The sample was then placed in the Carver press. Compaction was conducted at 6,000 psi. to yield a 10% reversion of the sodium tripolyphosphate, as compared to the unheated sample of Example 11, which exhibited no reversion. When 12,000 psi. were applied to a sample, the reversion was substantial, amounting to 50%.

The results indicate that the use of higher temperatures and/or compaction pressures, which cause a temperature rise, can induce substantial reversion of sodium tripolyphosphate to sodium ortho- and pyrophosphates, as evidenced by paper chromatography and titration (see Example 7).

Example 13

A pilot run was made by preparing a 1,000-pound blend of 20% of powdered sodium hydroxide and 80% of powdered, anhydrous sodium tripolyphosphate. The blend was added to a compaction system having counter-revolving rolls (not water-cooled rolls) 8 inches in diameter and 6 inches long (known as a Fitzpatrick chilsonator). The blend was carried up to the

compactor using an upwardly inclined and upwardly feeding screw conveyor, and was carried down to the rolls by a vertical screw feeder. The vertical screw feeder applied some pressure to the blend, but not enough to effect compaction of the blend to be
5 compacted. During the transfer and compaction by the rolls, it was noted that a great deal of heat was generated and the temperature of the product went up to 150°C. By paper chromatography analysis, it was found that 90% of the sodium tripolyphosphate reverted to sodium ortho- and pyrophosphates, as
10 evidenced by paper chromatography and titration (see Example 7).

A blend was also made in which 702 pounds of powdered, anhydrous sodium tripolyphosphate was admixed with 167 pounds of powdered sodium hydroxide, 79.2 pounds of sodium nitrite, and 52.5 pounds of sodium nitrate. This blend was also
15 put into the compaction system and a great amount of heat developed during conveying and compaction and substantial reversion occurred as above.

These results indicate the need or desire for temperature control of the blend before and during compaction
20 (see Example 9).

Example 14

A blend of 20% of powdered sodium hydroxide and 80% of powdered, anhydrous sodium tripolyphosphate was mechanically
25 mixed for a pilot run with a Fitzpatrick chilsonator having 10-inch diameter, counter-rotating, water-cooled rolls 7 inches long. The blend was fed into an overhead hopper, was fed through a horizontal screw conveyor less than 1 foot long to a downwardly feeding vertical screw feeder approximately 1 foot

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long. The product was then fed downwardly by the vertical screw feeder directly into the counter-rotating compaction rolls. Cooling water at a temperature of 68°F. was fed through the lower half of the vertical screw feeder and the
5 rolls. The horizontal screw conveyor revolved at 120 rpm., the vertical screw feed rotated at 1,200 rpm., and the rolls were revolved at 12 rpm.

Four different 30 or 35-pound batches (Samples 3A through 3D) were prepared and the results are shown in Table
10 17 below.

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Table 17

		Samples			
		3D	3A	3B	3C
5	Air Pressure to the Rolls (psi.)	12	16	30	35
	Oil Pressure to the Rolls (psi.)	400	600	1,000	1,100
	Total Pressure on the Rolls (psi.)	15,000	20,300	38,500	44,000
10	Specific Pressure on the 7-inch Rolls (pounds per linear inch)	2,143	2,900	5,500	6,286
15	Initial Temperature of the Phosphate-Sodium Hydroxide Mix (°F.)	74	74	74	74
20	Temperature of the Product Taken Immediately from the Compactor	78	84	90	86
	Temperature in the Drum 1½ hours later (°F.)	82	86	95	93
25	Degree of Compaction	Poor	Good	Good	Good
	Fragility of Compacted Product	Fragile	Fair	Hard	Hardest
30	Weight of Chips Cracked in the Laboratory (gms.)	181	227	234	235
	Material through a 5-Mesh Screen and on a 40-Mesh Screen (gms.)	149	191	203	207
35	Fines Through a 40-Mesh Screen (gms.)	32	36	32	28
	Percent Fines	18	16	14	12

In each case, about one minute was required for the compaction of the 30 or 35-pound batches. No visual evidence of reversion, as evidenced by lack of evolution of heat or

steam, could be detected in any of the four samples. This lack of reversion was confirmed by paper chromatography and titration (see Example 7).

The results shown in Table 17 indicate that by using the proper equipment and controlling the temperature of the blend before and during compaction, compacted granules can be made without substantial reversion of sodium tripolyphosphate.

Example 15

Another larger scale run was made using the Fitzpatrick chilsonator equipment used in the pilot run of Example 14, feeding 1,000 pounds of a blend of 80% of powdered, anhydrous sodium tripolyphosphate and 20% of powdered sodium hydroxide to the compaction rolls. The temperature of the blend was 97°F. Upon compaction at a pressure of 5,500 to 6,286 pounds per linear inch, the temperature of the resulting compacted granules rose to about 112°F. No substantial reversion of the sodium tripolyphosphate resulted, as determined by paper chromatography and titration (see Example 7).

The bulk of the compacted granules was placed in 250-pound drums (i.e., drums for storing and shipping 250 pounds of compacted granules). It was noted that within about 30 minutes, steam was evolved from the granules and the temperature of the granules reached about 280°F. Apparently sufficient heat was developed during compaction to trigger the reversion reaction.

The results appear to indicate the need or desirability for not feeding the blend to the rolls at a temperature above about 80°F., not allowing the compacted granules to reach a temperature very much above about 100°F. during or

after compaction, and cooling the compaction rolls.

Further, it is desirable not to treat the blend at a high humidity.

Example 16

5 One thousand pounds of an alkaline curing salt blend was made up by mechanically mixing the following proportions of materials:

	Powdered, Anhydrous Sodium Tripolyphosphate	8 Parts by Weight
10	Powdered Sodium Hydroxide	2 Parts by Weight
	Sodium Nitrite	0.8 Parts by Weight
	Sodium Nitrate	0.6 Parts by Weight
	Sodium Chloride	1.1 Parts by Weight

12.5 Pounds of the solid blend could be used to pre-
15 pare 50 gallons of a pickle liquor suitable for use as a 10% pump.

The initial temperature of the solid curing salt blend was 97°F. It was subjected to compaction with the chilsonator equipment described in Example 14, using a pressure
20 of 5,500 to 6,286 pounds per linear inch. Immediately after compaction, the temperature of the blend was 108°F. Small samples of the compacted blend showed no signs of reversion, as evidenced by the lack of evolution of heat or steam, and the product from the small compacted samples was put in 250-pound
25 bulk drums. No subsequent heat formation was noticed, and analyses using paper chromatography and titration (see Example 7) the following day showed no signs of substantial reversion.

The granules were then added to water and they dissolved readily. During the solution of these granules, it ap-

pears that the sodium hydroxide dissolves, developing heat, which rapidly disintegrates the granules, thus speeding the solution of the blend in the water.

A sample of the compacted blend was ground in a mortar with a pestle so that a major proportion of the blend would pass through a 5-mesh screen and be retained on a 40-mesh screen (the fines could be reblended and subjected to compaction). The amount of sodium nitrite and sodium hydroxide were determined by analyses as shown in Table 18 below.

10	<u>Table 18</u>	
	<u>Sodium Nitrite</u>	<u>Sodium Hydroxide</u>
	Theoretical:	6.40%
	16.0%	
	Found:	6.45%
	16.5%	

The analyses shown in Table 18 show that the compacted blend had the analyses as expected from the theoretical values.

The compaction bonded the sodium nitrite to the sodium tripolyphosphate and sodium hydroxide, and, as shown in Table 18A, each particle size fraction of the compacted material had comparable analyses.

	<u>Table 18A</u>	
		<u>% Sodium Nitrite</u>
	Theoretical	6.40
	Original Mechanical Mix	6.20
25	Compacted Material Cracked Through 40-Mesh Screen Through 5-Mesh Screen and on 40-Mesh Screen	6.06
		6.45

Example 17

A pickle liquor was prepared with the following com-

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position:

	Water	50 gallons (416.5 pounds)
	Sodium Chloride	71 pounds, 4 ounces
	Cane Sugar	25 pounds
5	Prague Powder	13 pounds, 12 ounces

4.2 Grams of calcium chloride were added to two 6.5-pound samples of the pickle liquor to increase the calcium content of the pickle liquor to about 500 ppm., which is often present in commercial lixated brines.

10 Two ounces of compacted Sample 3B of Example 14 (80% of powdered, anhydrous sodium tripolyphosphate and 20% of powdered sodium hydroxide), and 3.2 ounces of sodium tripolyphosphate were individually added to each one of the two 6.5-pound samples of pickle liquor (equivalent to 10 pounds of

15 Sample 3B and 16 pounds of sodium tripolyphosphate added to the 50 gallons of pickle liquor). Both pickle liquor samples were clear and had only a trace of precipitate, thus indicating the good solubility characteristics in the pickle liquor of Sample 3B, as well as sodium tripolyphosphate. The pickle liquors

20 were stored at 45°F. for 1 week.

Example 18

Several 300-pound lots of separate blends having (a) 80% of powdered, anhydrous sodium tripolyphosphate and 20% of powdered sodium hydroxide, and (b) the alkaline curing salt

25 blend of Example 16 were compacted using the counter-rotating compaction rolls of Allis Chalmers Manufacturing Company in Milwaukee, Wisconsin. The compaction rolls had a diameter of 30 inches, were 8 inches long, were not water-cooled, were rotated at 2 rpm., and applied a pressure of 15,000 pounds per

linear inch.

It was found that these rolls behave like the Fitzpatrick chilsonator rolls of Example 14 in these respects:

1. Both types of equipment can produce a compacted product suitable for the manufacture of satisfactory compacted granules; and

2. Both types of equipment develop enough heat of compaction in an ordinary atmosphere to trigger a violent reversion of the compressed product if the blend and equipment are improperly handled or used.

From this and previous tests and Example 21, it was learned that proper manufacturing procedures for producing compacted granules should involve the following:

(a) A low humidity environment at moderate temperatures (e.g., below about 80°F.);

(b) A blend similarly held at controlled temperatures (e.g., below about 80°F. prior to compaction);

(c) Not allowing the temperature of the blend to increase too high during compaction (e.g., below about 110°F.); and

(d) Maintaining the compacted product at a sufficiently low temperature prior to bulk packing so as to dissipate any latent heat which might trigger reversion.

Samples of the two compacted blends from Allis Chalmers Manufacturing Company were ground in a mortar with a pestle so that a major proportion of the blend would pass through a 5-mesh screen and be retained on a 40-mesh screen.

(The fines could be reblended and subjected to compaction.)

The analyses of the blends are set forth in Table 19 below.

Table 19

5	<u>Compacted Blend of 80% Sodium Tripolyphosphate and 20% Sodium Hydroxide</u>		<u>Compacted Blend of Example 16</u>	
	<u>Sodium Hydroxide</u>	<u>Nitrite</u>	<u>Sodium Hydroxide</u>	
Theoretical:	20%	6.4%	16.0%	
10 Found:	19%	6.2%	15.4%	

Example 19

Blends of Compositions "A", "B", and "C" were prepared as shown in Table 20 below.

Table 20

15	<u>Compositions</u>		
	<u>"A"</u>	<u>"B"</u>	<u>"C"</u>
Powdered, Anhydrous Sodium Tripolyphosphate	8 pounds	16 pounds	32 pounds
Powdered Sodium Hydroxide	2 pounds	2 pounds	2 pounds

20 The blends were placed in beakers immersed in a heated oil bath, and the blends were agitated with a paddle type stirrer for 30 minutes at the temperatures in Table 21 below.

Table 21

5	Compositions	Oil Bath Temperature	Maximum Temperature Rise of Composition	Time to Reach Maximum Temperature	Percent Reversion of Sodium Tripolyphosphate (Determined by Paper Chromatography)
	"A"	75°C. (167°F.)	8.0°C. (14°F.)	15 Minutes	50%
	"A"	47°C. (116°F.)	0.5°C. (1°F.)	25 Minutes	0
	"B"	76°C. (169°F.)	10.0°C. (18°F.)	18 Minutes	33%
	"B"	46°C. (115°F.)	1.0°C. (2°F.)	25 Minutes	0
10	"C"	75°C. (167°F.)	5.0°C. (9°F.)	23 Minutes	25-33%

The data in Table 21 above show that the sodium tri-
polyphosphate tends to revert to sodium ortho- and pyrophos-
phates as the level of heat increases. Within the limits
tested, the initiation of reversion does not appear to be de-
5 pendent upon the proportion of sodium hydroxide present in the
blend; however, if reversion does occur, the extent of the re-
version tends to increase with an increase of the proportion of
sodium hydroxide.

Composition "A" was modified by replacing the 80%
10 level of sodium tripolyphosphate with an equal level of two
different blends of phosphates which included more highly
molecularly dehydrated phosphates, and using 20% of sodium hy-
droxide. The resulting compositions, Compositions "D" and "E",
are shown in Table 22 below. The phosphate blend used for
15 Composition "D" is commercially available sodium hexametaphos-
phate and the phosphate blend of Composition "E" was "Brifisol"
of Joh. A. Benckiser G.m.b.H. The phosphates listed below so-
dium tripolyphosphate in Table 22 are more molecularly dehy-
drated.

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Table 22

		<u>Compositions</u>	
		<u>"D"</u>	<u>"E"</u>
	Sodium Orthophosphate	1.20%	0.48%
5	Sodium Pyrophosphate	2.24%	11.60%
	Sodium Tripolyphosphate	2.32%	25.28%
	Sodium Tetrapolyphosphate	4.56%	26.24%
	Sodium Trimetaphosphate	3.60%	15.52%
	Sodium Pentapolyphosphate	4.56%	0.88%
10	Sodium Hexapolyphosphate	3.84%	
	Sodium Hexametaphosphate	57.68%	
	Sodium Hydroxide	20.00%	20.00%
	TOTAL	100.00%	100.00%

Samples of Compositions "D" and "E" were placed in an oven and heated to the temperatures and for the time periods shown in Table 23 below. The degree of reversion of the particular phosphates subject to reversion was determined by paper chromatography.

Table 23

		Degree of Reversion of Certain of the Phos- phates in Compositions "D" and "E"	Percent Moisture Loss of Compositions "D" and "E"
<u>Heating Conditions</u>			
20	20 minutes at 200°C. (392°F.)	Extensive	6.0
25	15 minutes at 200°C. (392°F.)	Extensive	6.0
	15 minutes at 110°C. (230°F.)	Extensive	5.1

The stability of Compositions "D" and "E" under the conditions used in Example 9 was evaluated by paper chromatography and the data shown in Table 24 below were obtained.

Table 24

5	Compositions	Oil Bath Temperature	Maximum Temperature Rise of Sample	Time to Reach Maximum Temperature	Degree of Reversion of Certain of the Phosphates in Compo- sitions "D" and "E"
10	"D"	75°C. (167°F.)	Much Greater Than 50°C. (90°F.) (Steam Evolved)	7 Minutes	90% or more
	"D"	47°C. (116°F.)	3°C. (5°F.)	20 Minutes	0
	"E"	46°C. (115°F.)	2°C. (4°F.)	30 Minutes	Slight

The oil bath data in Table 24 above show that the more highly dehydrated phosphates are less heat stable than sodium tripolyphosphate. Thus, while 50% of the sodium tripolyphosphate reverted at 75°C. with mild heat evolution, as shown in Example 9, Composition "D" reverted violently and to a greater extent under the same conditions. Further, Composition "E" appears to be even more sensitive to heat and underwent some reversion at 47°C., whereas Composition "D" and the blend of Example 9 (not compacted and had 80% of sodium tripolyphosphate and 20% of sodium hydroxide) were stable at that temperature.

Example 20

The compacted blends of 80% sodium tripolyphosphate and 20% sodium hydroxide of Example 14 (Sample 3B), Example 15, and Example 18 (treated with rolls at Allis Chalmers Manufacturing Company), which were prepared at different times, were broken in a mortar with a pestle and were screened (a major proportion passed through a 5-mesh screen and was retained on a 40-mesh screen) to yield 5- to 40-mesh products. The products were stored in bulk using polyethylene liners in the drums at room temperatures for the periods shown in Table 25 below. The results shown in Table 25 below were obtained.

Table 25

	<u>Blends of Examples</u>	<u>Length of Storage</u>	<u>Degree of Reversion of Sodium Tripolyphosphate</u>
25	14 (Sample 3B)	30 days	None
	15	27 days	None
30	18 (Treated at Allis Chalmers Manufacturing Company)	20 days	None

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Table 25 above shows that no reversion was noted, as determined by paper chromatography or titration curves.

Example 21

5 A blend of 80% of sodium tripolyphosphate and 20% of sodium hydroxide was prepared. Approximately 250 pounds of the blend were stored in a polyethylene bag impervious to moisture and were kept at room temperature for stability testing.

Periodic evaluation of the blend by paper chromatography and titration curves revealed that the sodium tripolyphosphate did
10 not revert over a two-month period of storage.

After the two-month period of storage, accurately weighed 2.0-gram samples of the blend were exposed in two separate desiccators to atmospheres having a relative humidity, at room temperature, of 6% and 15%, respectively, as provided
15 by diluted sulfuric acid solutions (Handbook of Chemistry and Physics, 39th Edition, p. 2318). After an exposure time of three days, the samples were reweighed to determine moisture pickup and analyzed for reversion. The results are shown in Table 26.

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Table 26
Effect of Controlled Humidity on the Stability of Blend

5	% Relative Humidity of Environment	% Gain in Weight After 3 days' Exposure (Moisture)	Appearance	% Phosphate Composition Determined by Paper Chromatography			
				Sodium Orthophosphate	Sodium Pyrophosphate	Sodium Tripolyphosphate	
	6	3.8	Free-Flowing	0	5	95	
10	15	7.4	Clumped	20	40	40	

The data shown in Table 26 show that the sodium tripolyphosphate present in the blend, when exposed to 6% relative humidity for three days, did not undergo reversion and the blend had only the slight amount of sodium pyrophosphate content generally present in commercial sodium tripolyphosphate. On the other hand, when another portion of the blend was exposed to 15% relative humidity, 55% of the sodium tripolyphosphate reverted to a mixture of ortho- and pyrophosphates.

The foregoing detailed description has been given for clearness of understanding only, and no unnecessary limitations should be understood therefrom, as modifications will be obvious to those skilled in the art.

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SUPPLEMENTARY DISCLOSURE

This invention relates to a particulate composition suitable for use in meat curing media and having good solubility characteristics in a pickle liquor which comprises a composition substantially free of water and having about 70 to 94.5 parts by weight of alkali metal tripolyphosphate and about 30 to 5.5 parts by weight of alkali metal hydroxide, at least about 75% of said phosphate being capable of remaining non-reverted upon storage of said composition.

10 Further the invention relates to (a) a particulate composition suitable for use in meat curing media such as pickle liquor, which composition includes the combination, either as a mechanical mix of controlled stability or compacted granules of controlled stability, of particular amounts or proportions of alkali metal hydroxide and alkali metal tripolyphosphate, (b) a package having such composition within a moisture-impervious barrier, and (c) a method of making such compacted granules of the composition under controlled conditions and, if desired, placing such compacted granules within a moisture-impervious barrier.

20 The terms "sodium hydroxide" and "NaOH" as used herein include commercial sodium hydroxide such as commercial caustic soda. The term "alkali metal hydroxide" as used herein thus covers, among other things, commercial caustic soda.

The term "sodium tripolyphosphate" as used herein includes commercial grade sodium tripolyphosphate. The term "alkali metal tripolyphosphate" as used herein thus covers, among other things, commercial grade sodium tripolyphosphate.

It is known that phosphate salts function to increase

the water-binding capacity of meat in the presence or in the absence of other edible materials such as exemplified by sodium chloride, sugar, sodium nitrite, sodium nitrate, and acidic water-soluble antioxidant compounds having an enediol group, such as sodium erythorbate, sodium ascorbate and sodium isoascorbate (see U.S. Patents 2,739,899, 3,122,442, 3,139,347, 3,231,392 and 3,391,007).

We have discovered that unobvious advantages are obtained with particulate compositions having the new and unobvious combination of certain edible proportions of alkali metal tri-
10 polyphosphate and alkali metal hydroxide, as described below. Furthermore, we found that desirable benefits (e.g., good solubility in lixated brine) are obtained when such compositions do not introduce a substantial or undesired amount of reverted tri-
polyphosphate (orthophosphate or pyrophosphate) or reaction product of the alkali metal tripolyphosphate and alkali metal hydroxide into pickle liquor.

In its broadest aspects, our invention concerns particulate compositions for use in meat curing media having the
20 combination, either as a mechanical mix or granular blend or as compacted granules, of an alkali metal hydroxide such as sodium hydroxide and substantially non-reverted alkali metal tripolyphosphate such as sodium tripolyphosphate, either with or without other ingredients used in meat curing media (e.g., sugar, sodium, chloride, sodium nitrate, sodium nitrite, enediol compound such as sodium erythorbate, sodium ascorbate and sodium isoascorbate, etc.), including a nitric-oxide-producing curing medium, particularly in amounts of about 5.5 to 30 parts or

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percent by weight of alkali metal hydroxide and about 94.5 to 70 parts or percent by weight of alkali metal tripolyphosphate, or, about 10 to 20 parts or percent by weight of the hydroxide and about 90 to 80 parts or percent by the weight of the phosphate. These controlled proportions of the members of the combination allow the most effective use of the combination in meat curing media, so as to reduce the shrinkage of meat products and attendant purging of juices, to avoid the bleeding or salting out of phosphate on the surface of meat, and to obviate undesired taste qualities which can be found when excessive levels of either the hydroxide or phosphate compounds are used in treating meat.

The stability of the particulate composition is effected by excessive moisture and/or temperature conditions.

The particulate composition of our invention, regardless of whether it is a mechanical mix or compacted granules, must be substantially free of available moisture, so that a major portion or at least about 75% by weight of the alkali metal tripolyphosphate remains nonreverted or does not react with the alkali metal hydroxide upon storage of the composition. We have found, for example, that the available moisture content should be less than about 5% or, preferably, not more than about 2% by weight (e.g., based on the weight of the alkali metal tripolyphosphate and alkali metal hydroxide) of added, free, or available water, and the tripolyphosphate should be capable of remaining in such nonreverted state (e.g., in a moisture-impervious barrier such as a bag or drum stored at room temperature) until the composition is normally used under ordinary or con-

ventional commercial conditions (e.g., for at least about 2 months at room temperature).

We prefer that the particulate composition of our invention be free of other materials or quantities thereof which prevent the composition from having sufficiently good solubility characteristics in commercial pickle liquor, so that the liquor is substantially free of turbidity resulting, for example, from insoluble calcium precipitate formed from or with the composition and impurities found with some commercial grades of sodium chloride (commercial sodium chloride can have mineral impurities which provide calcium ions) used in pickle liquor.

When proper proportions of a selected amount of the combination (e.g., sodium hydroxide and sodium tripolyphosphate) was used by us in a pickle liquor pumped in ham, we found that less shrinkage resulted in the ham than when either sodium tripolyphosphate was used without sodium hydroxide, or when sodium hydroxide was used without the phosphate. When sodium hydroxide was used in a pickle liquor without sodium tripolyphosphate, there was more shrinkage in ham, despite the fact that the ham had a slightly higher pH, than resulted with the use of sodium tripolyphosphate without sodium hydroxide, and there was poorer utilization of nitrite (from pickle liquor) than occurred with the use of the phosphate without the sodium hydroxide.

We found that the use of a composition having or introducing too much sodium hydroxide, either with sodium tripolyphosphate or without it, caused slight bitterness in ham; similarly, the use of too much sodium tripolyphosphate without the sodium hydroxide provided some indication of a slight resi-

dual "phosphate" taste. Further, the use of too much tripolyphosphate can cause migration of the salt content, as well as residual phosphate, from the meat to the surface of meat in the form of white "snow" which imparts an undesirable surface taste and appearance.

When compositions having our combination of alkali metal tripolyphosphate and alkali metal hydroxide is formed by compaction, the product can be formed so as to be non-dusting, less hygroscopic, and of substantially uniform chemical content
10 for different gradations of size (although residual fines may require removal, reblending and compaction).

Several reversion problems, however, were encountered in attempting to compact compositions having alkali metal tripolyphosphate and alkali metal hydroxide.

We found that if the alkali metal tripolyphosphate-alkali metal hydroxide composition is subjected to too high a temperature or too much water or moisture before or during or after compaction, substantial reversion of the alkali metal tripolyphosphate is triggered or results. As one increases the
20 proportion of alkali metal hydroxide used, the alkali metal tripolyphosphate is more inclined to undergo increased reversion. The particular conditions which exist at one of these stages of manufacture or use effects the particular conditions which should or can exist at another stage and are interrelated and subject to some variation.

The temperature of the composition and the presence of water must be controlled, otherwise substantial reversion can result. Consequently, the amount of pressure used during

compaction and the type of compaction and related equipment used, the length of time during which compaction occurs, and the degree to which the presence of water is obviated, must be controlled.

When compaction is utilized in forming our compositions, a substantially uniform, dry mixture having substantially non-reverted alkali metal tripolyphosphate and alkali metal hydroxide should be used. The temperature and moisture content of that mixture must be controlled so as to avoid the initiation of substantial reversion of the phosphate. That mixture must be compacted under controlled temperature, moisture and compaction pressure conditions, thereby providing effective compaction and bonding together of the ingredients of the mixture without initiating or causing substantial reversion of the phosphate.

More specifically, in producing a substantially free-flowing, non-caking, non-dusting, compacted granular composition for use in meat curing media, we subject a substantially uniform, substantially dry, particulate mixture having, for example, about 70 to 94.5 parts or percent by weight of alkali metal tripolyphosphate and about 30 to 5.5 parts or percent by weight of alkali metal hydroxide to effective compaction pressure in the substantial absence of water under controlled compaction pressure and temperature conditions. This causes the effective and controlled compaction and bonding together of the ingredients of the mixture so that at least a major portion of the phosphate remains substantially non-reverted and produces a compacted, granular composition wherein at least a major portion or a substantial portion of the phosphate remains non-reverted upon

storage of the composition.

The substantially dry mixture which is to be subjected to compaction, for example, should have less than about 5% or not more than about 2% by weight added, available or free water or moisture (e.g., based on the weight of the tripolyphosphate and alkali metal hydroxide) and should not have or be subjected to a temperature above about 80°F., so that substantial reversion of the tripolyphosphate or substantial reaction of the phosphate with the alkali metal hydroxide is not initiated or triggered.

- 10 Compaction should be conducted under controlled conditions so that the temperature of the mixture does not exceed, for example, about 110°F. and its moisture content is not substantial (e.g., less than about 5% or not more than about 2% by weight added, available or free water), again, so that such reversion or re-action is not initiated or triggered.

- It will be apparent to those skilled in the art that the compaction necessary to accomplish the forming by granulation referred to above may be achieved in many ways. It is essential to form granules using compaction, although the
- 20 granules may be in the form, for example, of pellets, or even a cake which is subsequently crushed, but which results in a free-flowing or non-caking composition. The exact size and shape of the granules is not deemed to be important, provided the resulting product is free-flowing or non-caking under selected storage conditions.

Example 1 shows that the sodium tripolyphosphate present in a sodium tripolyphosphate-sodium hydroxide blend did not undergo substantial reversion during storage but did undergo

reversion in an atmosphere having too high a humidity.

Example 2 shows that compositions having sodium tri-
polyphosphate and sodium hydroxide and a substantial amount
of available moisture are unstable as evidenced by the reaction
of tripolyphosphate and sodium hydroxide and resulting reversion
of tripolyphosphate. This instability was more pronounced
when the proportion of moisture and/or sodium hydroxide present
in the composition is increased. The absence of substantial
water or the substantial absence of water affords the obtaining
10 of a substantially stable composition.

U. S. Patent 3,104,978 concerns a composition for
use in meat curing pickles allegedly comprising a partially
condensed, at least partially hydrated, amorphous alkali metal
phosphate composition prepared by reacting an alkali metal
tripolyphosphate with an alkali metal hydroxide capable of
at least hydrating the tripolyphosphate in the presence of
sufficient water to disperse the alkali metal compound and yield
a homogeneous product.

20 Although, generally, the maximum amount of water
in the aqueous solution of alkali metal compound is stated
in that patent as being equal to about half the amount necessary
to completely hydrate the tripolyphosphate (col. 2, lines 55-58),
preferably, water is added in a proportion equivalent to
between about 8% to about 13% by weight of the reaction
mixture, and, more particularly, in an upper portion of that
range (col. 2, lines 58-62).

The substantially homogeneous reaction product of

U. S. Patent No. 3,104,978 results from a substantially complete reaction (column 2, lines 66-68). The alkali metal hydroxide is used up during the reversion of the tripolyphosphate. In fact, Example 1 of that patent states in the last sentence thereof that chemical and physical tests of the product showed "no evidence of free sodium hydroxide".

U. S. Patent No. 3,104,978 also states that the reaction time needed to effect substantially complete reaction and to produce a substantially homogeneous product will vary with the type of mixing equipment employed, but generally the reaction may be completed in between two and about six hours, although greater or lesser reaction periods may be employed if desired (column 3, lines 11-18).

U. S. Patent No. 3,104,978 does not mention the packaging of the product in a moisture-impervious barrier--much less any need for doing so.

U. S. Patent No. 3,193,396 is concerned with uniformity of chemical composition, not with reversion of the phosphate. That patent, including column 3, lines 39-45, and Table 1 at the bottom of column 3, does not expressly disclose or point out, among other things, the following:

- (a) The combination, or the importance of having the combination, of alkali metal hydroxide and substantially non-reverted alkali metal tri-polyphosphate, either as a physical blend or compacted product, particularly in the proportions

disclosed herein. (It should be noted, for example, that Table I at the bottom of column 3 of the patent does not disclose that combination.);

(b) With blends of the combination, the presence of too much water or moisture, either added as such or resulting from humidity conditions and/or too high a temperature cause or trigger the tripolyphosphate to undergo substantial reversion in the presence of alkali metal hydroxide;

10 (c) Process conditions which include compaction and involve controlled temperature moisture or humidity, and compaction pressure conditions coordinated so as not to cause or trigger such reversion;

(d) The need for packaging the compositions of this patent application in a moisture-imperious barrier.

The Examples, which follow, are intended, among other things, to illustrate: the importance of having both an alkali metal tripolyphosphate and alkali metal hydroxide in our composition, particularly in the proportions mentioned above; the relationship of and results effected by varying levels of moisture, temperature, compaction conditions (if used), and/or storage conditions on the stability or reversion of various levels of the tripolyphosphate or the reaction between the alkali metal hydroxide and the tripolyphosphate; conditions which can be used and should or must be avoided.

20

All parts and percentages expressed in the Examples

are on a weight basis unless otherwise specified. Further, sodium tripolyphosphate mentioned in the Examples (unless indicated otherwise) is a commercial grade product and is in an anhydrous (substantially free of moisture and chemically bound water), powdered or granular form; the sodium hydroxide used (unless indicated otherwise) is also initially in an anhydrous (substantially free of moisture and chemically bound water), powdered or granular form; and, the blends that are compacted are substantially homogeneous or substantially uniformly blended before compaction.

The oil bath data in Table 24 show that the more highly dehydrated phosphates are less heat stable than sodium tripolyphosphate (limited levels of highly molecularly dehydrated phosphate, if desired, can be included in alkali metal hydroxide-alkali metal tripolyphosphate compositions, provided the resulting compositions have the required stability characteristics).

EXAMPLE 22

A blend of 80% of sodium tripolyphosphate and 20% of sodium hydroxide was prepared.

Approximately 250 pounds of a bulk blend of the sodium tripolyphosphate and sodium hydroxide was stored in polyethylene bags for purposes of stability tests. More specifically, approximately equal quantities of that bulk blend were divided into and stored in 5 separate polyethylene bags, and a separate 1 pound sample of the blend was stored in a tightly closed glass jar which was placed in the laboratory at room temperature.

Table 27 shows stability analyses which were made from the 1 pound sample at periodic intervals. Stability determina-

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tions were made to determine the degree of reversion of the sodium tripolyphosphate (to sodium pyrophosphate or sodium orthophosphate). The phosphate composition was determined by paper chromatography using appropriate standards or by titration curves (see Examples 7 and 8).

Table 27

Stability Determinations Made From 1 Pound Sample

Length of Storage	Phosphate Composition Determined by Paper Chromatography			Phosphate Composition Determined by Alternation of Titration Curves	
	% Sodium Tripolyphosphate	% Sodium Pyrophosphate	% Sodium Orthophosphate		
0 days	95	5	Trace	Normal Titration Pattern	
10 days	-	-	-	Substantially Unchanged	
14 days	95	5	Trace		
21 days	-	-	-	Substantially Unchanged	
23 days	95	5	Trace		
51 days	95	5	Trace		
64 days	95	5	Trace		

As is evident from Table 27 above, there was no substantial reversion of the sodium tripolyphosphate after 64 days of storage. Furthermore, after about 24 months of storage, the percent non-reverted sodium tripolyphosphate remained substantially unchanged as determined by paper chromatography.

Similarly, an analysis by paper chromatography of the bulk blend after about 11 months of storage showed that about 95% of the sodium tripolyphosphate remained unreverted.

After the 64-day period of storage mentioned above,
10 2-gram samples of the blend were exposed in two separate desiccators to atmospheres having a relative humidity, at room temperature, of 6% and 15%, respectively, as provided by diluted sulfuric acid solutions (Handbook of Chemistry and Physics, 39th Edition, p. 2318). After an exposure time of three days, the samples were reweighed to determine moisture pickup and analyzed for reversion. The results are shown in Table 26.

The data shown in Table 26 show that the sodium tripolyphosphate present in the blend, when exposed to 6% relative humidity for three days, did not undergo reversion and the blend
20 had only the slight amount of sodium pyrophosphate content generally present in commercial sodium tripolyphosphate. On the other hand, when another portion of the blend was exposed to 15% relative humidity, 55% of the sodium tripolyphosphate reverted to a mixture of ortho- and pyrophosphates.

EXAMPLE 23

The following tests were conducted for the purpose of evaluating sodium tripolyphosphate-sodium hydroxide compositions without added water and with varying amounts of water.

In the tests, the same proportions of the same materials disclosed in Example 1 of U.S. Patent 3,104,978 to Eugene V. Elder, Jr. were used, and that Example was also changed by omitting added water from the composition. Furthermore, the amounts of sodium tripolyphosphate and sodium hydroxide were varied, both without and with varying amounts of added water.

A mixed blend of the following materials was prepared:

Composition "A-1" (12.5% Added Water)

	Sodium Tripolyphosphate	92.3 grams
10	Sodium hydroxide	7.7 grams
	Water	14.3 grams

The foregoing weight proportions conform with the weight (total) proportions of the same ingredients used in Example 1 of U.S. Patent 3,104,978.

The blend of Composition "A-1", as well as the other Compositions mentioned later below, was divided into about equal parts, all but one of which parts were placed in tightly sealed jars at room temperature, the remaining one part was analyzed for reversion on the day it was prepared. Stability studies
20 were made of all samples to determine the degree of reversion of the sodium tripolyphosphate (to sodium ortho- or pyrophosphates).

The free or unreacted sodium hydroxide of Composition "A-1", as well as other Compositions mentioned later below, was determined (Tables 29, 30, 31, 32, 34 and 35) by extracting a 1-gram sample of the blend with 100 ml. of ethyl alcohol, and the extract was titrated with 0.1N sulfuric acid to a pH of about 7.0. This method determines the sodium hydroxide content, since the phosphates, sodium tripolyphosphate and its reversion

products (e.g., sodium ortho- and pyrophosphates), are not soluble in alcohol. The moisture content determinations shown in Tables 28 and 35 below, were determined by drying a sample of the blend for 6 hours in a vacuum oven at 50 C. (112 F.). The phosphate composition of the Composition "A-1", as well as the other Compositions shown below, was determined (Tables 29, 30, 31, 32, 34, 35 and 36) by paper chromatography as was used in Example 8.

Table 28 below shows the analysis of the moisture present in Composition "A-1" on the day it was prepared.

Table 28

Moisture Content of Composition "A-1" (12.5% Added Water)

	<u>Added Water</u>	<u>Determined by Analysis</u>
Moisture	12.5%	13.0%

Table 29 below shows the analyses of the stability of Composition "A-1" at periodic intervals. Fresh jars were opened to eliminate or obviate the possibility of breakdown or reversion due to frequent handling; the same procedure was used in the other stability determinations shown later below. The results were as shown in Table 29 below.

Table 29

Stability of Composition "A-1" (12.5% Added Water)

<u>Length of Storage at Room Temperature</u>	<u>pH (1% Solution)</u>	<u>% NaOH (Alcohol- Soluble)</u>	<u>% Sodium Tripolyphosphate which Reverted</u>
0 days	11.40	5.1*	0**
3 days	11.35	4.4	5
5 days	11.30	3.4	-

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7 days	11.35	1.8	20
10 days	11.20	1.2	35
14 days	11.15	0.6	35
21 days	11.00	0.1	35

* Initial sodium hydroxide content was 6.7%.

** Initial sodium tripolyphosphate content was 80.8%.

It is evident from Table 29 above that after 5 days of storage, about 49.3% by weight of the sodium hydroxide from Composition "A-1", which was made with 12.5% by weight of added water, had reacted with sodium tripolyphosphate initially used and this resulted in reversion of tripolyphosphate. After 7 days of storage, about 73.1% by weight of the sodium hydroxide had reacted and about 20% by weight of the tripolyphosphate had reverted. Furthermore, after 10 days of storage, about 82.1% by weight of the sodium hydroxide had reacted and about 35% by weight of the tripolyphosphate had reverted.

A mixed blend of the following materials was prepared:

Composition "A-2" (No Added Water)

20	Sodium Tripolyphosphate	92.3 grams
	Sodium Hydroxide	7.7 grams

The foregoing proportion of sodium tripolyphosphate and sodium hydroxide conforms with the proportion used in Example 1 of U.S. Patent 3,104,978 and Composition "A-1", but no water was added.

Table 30 below shows the analyses of the stability of Composition "A-2" at periodic intervals.

Table 30Stability of Composition "A-2" (No Added Water)

	<u>Length of Storage at Room Temperature</u>	<u>pH (1% Solution)</u>	<u>% NaOH (Alcohol- Soluble)</u>	<u>%Sodium Tripolyphosphate which Reverted</u>
	0 days	11.40	7.7*	0**
	3 days	11.45	6.9	0
	5 days	11.50	7.2	0
	7 days	11.60	6.4	0
10	10 days	11.55	6.5	0
	14 days	11.55	5.9	0
	21 days	11.55	6.9	0

* Initial sodium hydroxide content was 7.7%.

** Initial sodium tripolyphosphate content was 92.3%.

It is evident from Table 30 above that after 21 days of storage, the sodium tripolyphosphate remained substantially non-reverted, the pH remained substantially unchanged, and the sodium hydroxide remained substantially unchanged.

A mixed blend of the following materials was prepared:

20 Composition "B-1" (12.5% Added Water)

Sodium Tripolyphosphate	70.0 grams
Sodium Hydroxide	30.0 grams
Water	14.3 grams

The above proportion of sodium tripolyphosphate and sodium hydroxide (without added water) is disclosed in said application.

Table 31 below shows the analyses of the stability of Composition "B-1" at periodic intervals.

Table 31Stability of Composition "B-1" (12.5% Added Water)

	<u>Length of Storage at Room Temperature</u>	<u>pH (1% Solution)</u>	<u>% NaOH (Alcohol- Soluble)</u>	<u>% Sodium Tripolyphosphate which Reverted</u>
	0 days	11.85	24.8*	0**
	3 days	11.70	20.8	30
	5 days	11.75	18.9	-
	7 days	11.85	16.3	55
10	10 days	11.75	15.5	55
	14 days	11.80	14.3	65
	21 days	11.75	14.1	75

* Initial sodium hydroxide content was 26.2%.

** Initial sodium tripolyphosphate content was 61.2%.

It is evident from Table 31 above that after 3 days of storage, about 20.5% by weight of the sodium hydroxide from Composition "B-1", which was made with 12.5% by weight of added water, had reacted with sodium tripolyphosphate initially used and this resulted in reversion of about 30% by weight of the tripolyphosphate. After 5 days of storage, about 27.9% by weight of the sodium hydroxide had reacted. Furthermore, after 7 days of storage, about 37.8% by weight of the sodium hydroxide had reacted and about 55% by weight of the tripolyphosphate had reverted. The presence of an increased proportion of sodium hydroxide in the blend in the presence of moisture tends to increase the reversion of sodium tripolyphosphate.

A mixed blend of the following materials was prepared:

Composition "B-2" (No Added Water)

Sodium Tripolyphosphate 70.0 grams

Sodium Hydroxide 30.0 grams

The foregoing weight proportion of materials conforms with the proportion of sodium tripolyphosphate and sodium hydroxide used in Composition "B-1", but there was no added water.

Table 32 below shows the analyses of the stability of Composition "B-2" at periodic intervals.

Table 32

Stability of Composition "B-2" (No Added Water)

10	Lenght of Storage at Room Temperature	pH (1% Solution)	% NaOH (Alcohol- Soluble)	% Sodium Tripolyphosphate which Reverted
	0 days	11.9	31.1*	0**
	3 days	11.8	30.6	0
	5 days	11.8	29.4	-
	7 days	11.9	26.7	0
	10 days	11.9	29.5	0
	14 days	11.9	28.8	0
	21 days	11.9	28.1	0

* Initial sodium hydroxide content was 30%.

** Initial sodium tripolyphosphate content was 70%.

20 It is evident from Table 32 above that after 21 days of storage of Composition "B-2", with no added water, there was no substantial reversion of sodium tripolyphosphate despite the presence of an increased proportion of sodium hydroxide.

A mixed blend of the following materials was prepared:

Composition "C-1" (12.5% Added Water)

Sodium Tripolyphosphate	94.5 grams
Sodium Hydroxide	5.5 grams
Water	14.3 grams

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The above proportion of sodium tripolyphosphate and sodium hydroxide (without added water) is disclosed in said application.

Table 33 below shows the analysis of the moisture present in Composition "C-1" on the day it was prepared.

Table 33

Moisture Content of Composition
"C-1" (12.5% Added Water)

	<u>Added Water</u>	<u>Determined by Analysis</u>
10 Moisture	12.5%	11.35%

Table 34 below shows the analyses of the stability of Composition "C-1" at periodic intervals.

Table 34

Stability of Composition "C-1" (12.5% Added Water)

	<u>Length of Storage at Room Temperature</u>	<u>pH (1% Solution)</u>	<u>% NaOH (Alcohol- Soluble)</u>	<u>% Sodium Tripolyphosphate which Reverted</u>
	0 days	11.35	3.7*	0**
	3 days	11.20	2.4	0
20	5 days	11.15	2.1	-
	7 days	11.20	0.9	5
	10 days	11.10	0.8	25
	14 days	11.10	0.5	30
	21 days	11.10	0.2	30

* Initial sodium hydroxide content was 4.8%.

** Initial sodium tripolyphosphate content was 82.7%.

It is evident from Table 34 above that after 5 days of storage, about 56.3% by weight of the sodium hydroxide from Composition "C-1", which was made with 12.5% by weight of added water, had reacted with the sodium tripolyphosphate

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initially used; this resulted in reversion of tripolyphosphate. After 10 days of storage, about 83.3% by weight of the sodium hydroxide had reacted and about 25% by weight of the tripolyphosphate had reverted. Furthermore, after 14 days of storage, about 89.6% by weight of the sodium hydroxide had reacted and about 30% by weight of the tripolyphosphate had reverted.

A mixed blend of the following materials was prepared:

Composition "C-2" (No Added Water)

	Sodium Tripolyphosphate	94.5 grams
10	Sodium Hydroxide	5.5 grams

The foregoing weight proportion of materials conforms with the proportion of sodium tripolyphosphate and sodium hydroxide used in Composition "C-1", but no water was added.

Table 35 below shows the analyses of the stability of Composition "C-2" at periodic intervals.

Table 35

Stability of Composition "C-2" (No Added Water)

20	<u>Length of Storage at Room Temperature</u>	<u>pH (1% Solution)</u>	<u>% NaOH (Alcohol-Soluble)</u>	<u>% Sodium Tripolyphosphate which Reverted</u>
	0 days	11.50	4.7*	0**
	3 days	11.35	4.7	0
	5 days	11.35	5.0	-
	7 days	11.45	3.9	0
	10 days	11.45	4.0	0
	14 days	11.40	3.9	0
	21 days	11.45	4.4	0

* Initial sodium hydroxide content was 5.5%.

** Initial sodium tripolyphosphate content was 94.5%.

It is evident from Table 35 above that after 21 days of storage of Composition "C-2", which was made with no added water, the sodium tripolyphosphate thereof remained substantially non-reverted.

A mixed blend of the following materials was prepared:

Composition "D-1" (No Added Water)

Sodium Tripolyphosphate 80 grams

Sodium Hydroxide 20 grams

Compositions "D-2", "D-3" and "D-4" were prepared
10 with the same proportion or ratio of sodium tripolyphosphate and sodium hydroxide used in Composition "D-1", except 2%, 5% and 12.5% by weight of water (based on the total weight of the respective compositions), respectively, were added to and mixed with these materials.

Table 36 below shows the analyses of the stability of Compositions "D-1", "D-2", "D-3" and "D-4" at periodic intervals.

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Table 36

Stability of Compositions "D-1", "D-2", "D-3" and "D-4"

Length of Storage at Room Temperature	"D-1" (No Added Water)				"D-2" (2% Added Water)				"D-3" (5% Added Water)				"D-4" (12.5% Added Water)			
	% Sodium Tripoly- phosphate which Reverted				% Sodium Tripoly- phosphate which Reverted				% Sodium Tripoly- phosphate which Reverted				% Sodium Tripoly- phosphate which Reverted			
0 days	0	1	0	2	0	2	0	3	0	2	0	3	0	2	0	4
3 days	0	0	0	0	0	0	0	20	0	0	0	20	0	0	0	35
7 days	0	0	0	5	0	5	0	30	0	5	0	30	0	5	0	45
10 days	0	0	0	-	0	-	0	-	0	-	0	-	0	-	0	-
14 days	0	0	0	5	0	5	0	35	0	5	0	35	0	5	0	50
15 days	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-

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1. Initial sodium tripolyphosphate content was 80%.
2. Initial sodium tripolyphosphate content was 78.4%.
3. Initial sodium tripolyphosphate content was 76%.
4. Initial sodium tripolyphosphate content was 69.9%.

It is evident from Table 36 above that after 15 days of storage, the sodium tripolyphosphate Composition "D-1", with no added water, remained substantially non-reverted. Substantially the same results were obtained after 15 days of storage with Composition "D-2", which had 2% by weight of water added during its initial preparation. After 3 days of storage of Composition "D-3", which was made with 5% by weight of added water, about 20% by weight of the sodium tripolyphosphate had reverted; after 7 days of storage, about 30% by weight of the sodium tripolyphosphate reverted; after about 14 days of storage, about 35% by weight of the sodium tripolyphosphate reverted. With Composition "D-4", which was prepared with 12.5% by weight of water, after 3 days of storage, about 35% by weight of the sodium tripolyphosphate reverted.

The foregoing detailed description has been given for clearness of understanding only, and no unnecessary limitations should be understood therefrom, as modifications will be obvious to those skilled in the art.

The embodiments of the invention in which an exclusive property or privilege is claimed are defined as follows:

1. A particulate composition suitable for use in meat curing which comprises substantially non-reverted alkali metal tripolyphosphate and alkali metal hydroxide.
2. The composition of claim 1 which includes alkali metal nitrate, alkali metal nitrite or admixtures thereof.
3. The composition of claim 1 which includes sodium chloride and alkali metal nitrate, alkali metal nitrite or admixtures thereof.
4. The composition of claim 1, 2 or 3 wherein the alkali metal tripolyphosphate and alkali metal hydroxide are sodium tripolyphosphate and sodium hydroxide, respectively.
5. The composition of claim 1, 2 or 3 wherein the composition has about 94.5 to 70 parts by weight of alkali metal tripolyphosphate and about 5.5 to 30 parts by weight of alkali metal hydroxide.
6. The composition of claim 1, 2 or 3 wherein the composition has about 90 to 80 parts by weight of alkali metal tripolyphosphate and 10 to 20 parts by weight of alkali metal hydroxide.
7. The composition of claim 1 which includes sodium chloride.
8. The composition of claim 7 wherein the alkali metal tripolyphosphate and alkali metal hydroxide are sodium tripolyphosphate and sodium hydroxide, respectively.
9. The composition of claim 7 wherein the composition has about 94.5 to 70 parts by weight of alkali metal tripolyphosphate and about 5.5 to 30 parts by weight of alkali metal hydroxide.

10. The composition of claim 7, 8 or 9 wherein the composition has about 90 to 80 parts by weight of alkali metal tripolyphosphate and 10 to 20 parts by weight of alkali metal hydroxide.

11. A particulate composition suitable for use in meat curing which comprises substantially free-flowing, non-caking, non-dusting, quick dissolving, chemically uniform granules having about 94.5 to 70 parts by weight of substantially non-reverted sodium tripolyphosphate bonded to about 5.5 to 30 parts by weight of sodium hydroxide.

12. The composition of claim 11 wherein the granules have about 90 to 80 parts by weight of substantially non-reverted sodium tripolyphosphate compaction bonded to about 10 to 20 parts by weight of sodium hydroxide.

13. A method of producing a composition suitable for use in meat curing media which comprises forming a substantially a uniform, particulate mixture having substantially non-reverted alkali metal tripolyphosphate and alkali metal hydroxide under controlled temperature and moisture conditions to avoid the initiation of occurrence of substantial reversion of said phosphate.

14. The method of claim 13 wherein the mixture is placed within a substantially moisture impervious barrier to produce a composition wherein said phosphate does not undergo substantial reversion.

15. A method of producing substantially non-caking, non-dusting compositions suitable for use in meat curing media having granules which comprises obtaining a substantially uniform, dry mixture having substantially non-reverted alkali metal tripolyphosphate and alkali metal hydroxide and controlling the temperature and moisture content of said mixture

to avoid the initiation of substantial reversion of said phosphate, and compacting said mixture under controlled temperature, moisture and compaction pressure conditions to cause effective compaction and bonding together of the ingredients of said mixture without initiating substantial reversion of said phosphate, to produce a composition having granules wherein said phosphate has not undergone substantial reversion.

16. The method of claim 15 wherein the mixture includes sodium chloride.

17. The method of claim 13, 14, or 15 wherein the mixture includes alkali metal nitrate, alkali metal nitrite or admixtures thereof.

18. The method of claim 13, 14, or 15 wherein the mixture includes sodium chloride and alkali metal nitrate, alkali metal nitrite or admixtures thereof.

19. The method of claim 16 wherein the mixture is at a temperature not exceeding about 80°F. prior to compaction, does not reach a temperature exceeding about 110°F. during compaction, and is packaged under temperature and moisture conditions wherein substantial reversion does not result.

20. The method of claim 16 or claim 19 wherein compacting is effected by water-cooled compaction rolls.

21. The method of claim 14, 15 or 16 wherein the alkali metal tripolyphosphate and alkali metal hydroxide are sodium tri-polyphosphate and sodium hydroxide, respectively.

22. The method of claim 14, 15 or 16 wherein the mixture has about 94.5 to 70 parts by weight of alkali metal tripolyphosphate and about 5.5 to 30 parts by weight of alkali metal hydroxide.

23. The method of claim 14, 15 or 16 wherein the mixture has about 90 to 80 parts by weight of alkali metal

tripolyphosphate and 10 to 20 parts by weight of alkali metal hydroxide.

24. A method of producing substantially free-flowing, non-caking, non-dusting, quick-dissolving, uniform compositions having granules and suitable for use in meat curing media, which method comprises obtaining a substantially uniform, dry mixture having about 94.5 to 70 parts by weight of substantially non-reverted sodium tripolyphosphate and about 5.5 to 30 parts by weight of sodium hydroxide and controlling the temperature and moisture content of said mixture to avoid the initiation of substantial reversion of said phosphate, and compacting said mixture under controlled temperature, moisture and compaction pressure conditions to cause effective compaction and bonding together of the ingredients of said mixture without initiating substantial reversion of said phosphate, to produce a composition having granules wherein said phosphate has not undergone substantial reversion.

CLAIMS BASED ON SUPPLEMENTARY DISCLOSURE

25. A particulate composition suitable for use in meat curing media and having good solubility characteristics in a pickle liquor which comprises a composition substantially free of water and having about 70 to 94.5 parts by weight of alkali metal tripolyphosphate and about 30 to 5.5 parts by weight of alkali metal hydroxide, at least about 75% of said phosphate being capable of remaining non-reverted upon storage of said composition.

26. The composition of claim 25 wherein said alkali metal tripolyphosphate is sodium tripolyphosphate and said alkali metal hydroxide is sodium hydroxide and said composition has less than about 5% by weight moisture.

27. The composition of claim 25 or 26 wherein said composition includes at least one member selected from the group consisting of sodium chloride, sugar, alkali metal nitrite, alkali metal nitrate, and anti-oxidant compound having an enediol group.

28. The composition of claim 26 wherein at least about 75% of said phosphate being capable of remaining non-reverted upon storage of said composition in a moisture-imperious barrier.

29. The composition of claim 26 wherein said composition includes at least one member selected from the group consisting of sodium chloride, sugar, alkali metal nitrite, alkali metal nitrate, and anti-oxidant compound having an enediol group and at least about 75% of said phosphate being capable of remaining non-reverted upon storage of said composition in a moisture-imperious barrier.

30. The composition of claim 29 wherein at least about 75% of said phosphate is capable of remaining non-reverted upon storage of said composition in a moisture-impervious barrier for at least about 2 months.

31. The composition of claim 25 or 26 which is substantially non-caking, free-flowing, non-dusting, quick-dissolving and compacted and in the form of substantially chemically uniform and non-fragile granules.

32. The composition of claim 25 or 26 wherein it is within a substantially moisture-impervious barrier in the form of a package.

33. The composition of claim 25 or 26 wherein said composition includes at least one member selected from the group consisting of sodium chloride, sugar, alkali metal nitrite, alkali metal nitrate, and anti-oxidant compound having an enediol group and is within a substantially moisture-impervious barrier in the form of a package.

34. The composition of claim 25 or 26 which is substantially non-caking, free-flowing, non-dusting, quick-dissolving and compacted and in the form of substantially chemically uniform and non-fragile granules and is within a substantially moisture-impervious barrier in the form of a package.

35. The composition of claim 25 or 26 wherein said alkali metal triphosphosphate is sodium triphosphosphate and said alkali metal hydroxide is sodium hydroxide and said composition has less than about 2% by weight moisture and wherein said composition includes at least one member selected from the group consisting of sodium chloride, sugar, alkali metal nitrite, alkali metal nitrate, and anti-oxidant compound having an enediol

group and is within a substantially moisture-impervious barrier in the form of a package.

36. The composition of claim 25 or 26 wherein said alkali metal tripolyphosphate is sodium tripolyphosphate and said alkali metal hydroxide is sodium hydroxide and said composition has less than about 2% by weight moisture and is substantially non-caking, free-flowing, non-dusting, quick-dissolving and compacted and in the form of substantially chemically uniform and non-fragile granules and is within a substantially moisture-impervious barrier in the form of a package.

37. The method of producing the composition of claim 25 or 26 which method comprises subjecting a substantially uniform, substantially dry, particulate mixture of the alkali metal tripolyphosphate and the alkali metal hydroxide to compaction pressure in the substantial absence of water and under controlled compaction pressure and temperature conditions to cause effective, controlled compaction and bonding together of the ingredients of said mixture so that a major portion of said phosphate remains non-reverted, to produce said compacted granular composition wherein a major portion of said phosphate remains non-reverted upon storage of said composition.

38. The method of claim 37 wherein said alkali metal tripolyphosphate is sodium tripolyphosphate and said alkali metal hydroxide is sodium hydroxide.

39. The method of claim 37 or 38 comprising controlling the temperature of said mixture so that the temperature of said mixture does not exceed about 80°F. and substantial reversion of said phosphate is not initiated, and compacting said mixture in the substantial absence of water and under

controlled compaction pressure and temperature conditions to cause effective compaction and bonding together to the ingredients of said mixture so that the temperature of said mixture does not exceed about 110°F,

40. The method of claim 37 or 38 comprising placing said compacted granular composition within a moisture-impervious barrier, a major portion of said phosphate remaining non-reverted upon storage of said composition within said barrier.

41. The method of claim 37 or 38 comprising controlling the temperature of said mixture so that the temperature of said mixture does not exceed about 80°F. and substantial reversion of said phosphate is not initiated, and compacting said mixture in the substantial absence of water and under controlled compaction pressure and temperature conditions to cause effective compaction and bonding together to the ingredients of said mixture so that the temperature of said mixture does not exceed about 110°F. and placing said compacted granular composition within a moisture-impervious barrier, a major portion of said phosphate remaining non-reverted upon storage of said composition within said barrier.